
SIGILLVM · VNIVERSITATIS · CALIFORNIENSIS
LVX
FIAT
MDCCCLXVIII
EX LIBRIS

7. 26-42
112-187

Bulletin 210

DEPARTMENT OF THE INTERIOR
ALBERT B. FALL, SECRETARY
BUREAU OF MINES
H. FOSTER BAIN, DIRECTOR

OIL-SHALE
AN HISTORICAL, TECHNICAL, AND ECONOMIC STUDY

BY

MARTIN J. GAVIN

STATE OF COLORADO CO-OPERATIVE OIL-SHALE INVESTIGATIONS

(This bulletin was printed by the State of Colorado, as part of the co-operative agreement with the Bureau of Mines for the investigation of oil-shales.)

DENVER
THE BRADFORD-ROBINSON PRINTING CO.
1922

The Bureau of Mines, in carrying out one of the provisions of its organic act—to desseminate information concerning investigations made—prints a limited free edition of each of its publications.

The State of Colorado, co-operating with the Bureau of Mines in its oil-shale investigations, has provided the funds for the publication of the first edition of this bulletin, that it might be available sooner than if printed by the Government Printing Office. When this edition is exhausted, a second edition will be printed by the Government Printing Office, Washington, D. C.

The general law under which publications are distributed prohibits the giving of more than one copy of a publication to one person. Copies of this bulletin can be obtained from U. S. Bureau of Mines, Boulder, Colorado.

First edition, July, 1922.

TYPICAL OIL-SHALE FORMATION NEAR DEBEQUE, COLO.
(Darker and more resistant beds are richer shales)

CONTENTS.

APPENDIX.

TABLES.

ILLUSTRATIONS.

FOREWORD.

The State of Colorado, among its resources of potential importance, considers its vast oil-shale deposits of particular value and the basis of a future great industry that ultimately will place Colorado in the front rank of the oil producing States of the Union. It affords me much gratification that the United States Bureau of Mines has so generously co-operated with the State of Colorado in working out the solution of some fundamental problems of the coming industry and in presenting the results of its studies to the public.

OLIVER H. SHOUP,
Governor.

PREFACE.

In January, 1920, the State of Colorado and the United States Bureau of Mines entered into a co-operative agreement for the conduct of laboratory investigations on the oil-shales of Colorado. Under this agreement a laboratory has been installed and equipped at the State University, Boulder, Colorado, and a research staff organized. It is the primary purpose of the investigational work to determine the most favorable conditions of retorting Colorado oil-shales to yield the most of the best products from them.

Several papers dealing with the program of the investigations and with some of the work already accomplished, have already been published in mimeographed form by the Bureau of Mines, and it is the purpose of the Bureau and State to continue publishing short reports as frequently as material becomes available.

This bulletin represents the assembled results of the investigations up to the present, together with material of a general nature necessary in a well-rounded presentation of the present state of knowledge of the subject.

I take pleasure in acknowledging, not only the co-operation of the State of Colorado, already referred to above, but also its arranging for the publication of this bulletin, which permits it to be issued at a much earlier date than would otherwise have been possible.

H. FOSTER BAIN.

OIL-SHALE
AN HISTORICAL, TECHNICAL, AND ECONOMIC STUDY

By Martin J. Gavin.

INTRODUCTION.

The results of investigations of the oil-shale resources of the United States were first published by the United States Geological Survey, in 1915.[1] Other reports[2] have followed.

These reports, investigations by private individuals, and the beginning of the development of an oil-shale industry in this country have created considerable interest in the possibilities of these shales as a commercial source of hydrocarbon oils. The Bureau of Mines has appreciated these possibilities, and since 1916 one or more of its engineers have given all or part of their time to investigations relating to the treatment of oil-shales. A short pamphlet[3] containing general information on the possibilities of oil shale in this country, particularly the shales of the Rocky Mountain District, has already been published. This pamphlet gives methods for assaying the shales for oil and ammonia, and contains a bibliography for the use of those desiring to search the literature.

Further investigations of oil-shales and the oil-shale industry, both in this country and in Scotland, where shales are being treated commercially, have made it possible to present the subject in greater detail. Since most of the published information on oil-shale is scattered through a great many technical journals and treatises on petroleum and mineral oils, this bulletin summarizes pertinent facts of the published literature, and in addition contains information that has been obtained by the Bureau of Mines in its investigations.

1 Woodruff, E. G., and Day, D. T., Oil-shale of northwestern Colorado and northeastern Utah; U. S. Geol. Survey, Bull. 581, 1915, pp. 1-22.

2 Winchester, D. E., Oil-shale in northwestern Colorado and adjacent areas; U. S. Geol. Survey, Bull. 641, 1917, pp. 139-198. ...., Oil-shale of the Uinta Basin, northeastern Utah, and results of dry distillation of miscellaneous shale samples: U. S. Geol. Survey, Bull. 691, 1918, pp. 27-55.

Ashley, G. H., Oil resources of black shales of the Eastern United States: U. S. Geol. Survey, Bull. 641, 1917, pp. 311-324.

Bowen, C. F., Phosphatic oil-shales near Dell and Dillon, Beaverhead County, Montana: U. S. Geol. Survey, Bull. 661, 1918, pp. 315-320.

Condit, D. D., Oil-shale in western Montana, southeastern Idaho and adjacent parts of Wyoming and Utah: U. S. Geol. Survey, Bull. 711B, 1919, 26 pages.

3 Gavin, M. J.; Hill, H. H., and Perdew, W. E., Notes on the oil-shale industry with particular reference to the Rocky Mountain District: U. S. Bureau of Mines, Reports of Investigations, Serial No. 2256, 1921, 36 pages.

Throughout the bulletin, unless stated otherwise, a ton is 2,000 pounds, and a gallon 231 cubic inches (U. S. gallon). Likewise, a barrel is 42 gallons of 231 cubic inches each.

ACKNOWLEDGMENTS.

It is not possible to acknowledge individually the services of the many people who have aided the preparation of this bulletin, but the author wishes to extend to them thanks.

Acknowledgment is due particularly, however, to Sir John Cadman, former director of His Majesty's Petroleum Executive, London, without whose aid much of the information pertaining to the Scotch oil-shale industry could not have been obtained; to Messrs. William Frazer, H. R. J. Conacher, James Bryson and A. C. Thomson, of Scottish Oils, Limited, Glasgow, Scotland; to Mr. H. M. Cadell of Linlithgow, Scotland, for much information regarding the Scotch industry; and to Mr. R. H. Cambage, Department of Mines, New South Wales, for information regarding the industry in Australia. Special acknowledgment is due to Mr. J. O. Lewis, former chief petroleum technologist of the Bureau of Mines; to Mr. A. W. Ambrose, present chief petroleum technologist of the Bureau, and to Mr. W. E. Perdew, Union Petroleum Co., Philadelphia, for valuable data, suggestions and criticisms.

The bibliography was prepared by Miss E. H. Burroughs, bibliographer of the Bureau of Mines.

Acknowledgment is also extended to the author's colleagues in the Bureau of Mines for assistance in the preparation and correction of the manuscript, particularly to Messrs. L. C. Karrick, J. J. Jakowsky, and L. H. Sharp. After the manuscript was completed, it was read and constructively criticized by Messrs. N. A. C. Smith and A. W. Ambrose of the Bureau of Mines; James Bryson, A. C. Thomson and H. R. J. Conacher of Scottish Oils, Ltd., and by Mr. Perdew of the Union Petroleum Company. The drawings were prepared by Mr. J. D. Shumate and Mr. J. J. Jakowsky, of the Bureau of Mines.

THE PRESENT STATUS OF THE PETROLEUM INDUSTRY IN THE UNITED STATES.

Only within the last few years have the American public, the Government and the American oil companies appreciated the gravity of the situation that will result from our oil supply becoming so depleted as to be inadequate to meet demands. The situation as regards petroleum is nowise different from that of any other natural resource. When the supply of any resource is plentiful and easily obtainable, little attention is paid to conservation or efficient utilization, but when the supply begins to fail, conservation begins to be applied and possible substitutes are sought.

A comparatively few years ago petroleum and its products, being plentiful and easily obtained, were produced and utilized with little regard to conservation. Millions of barrels of petroleum and billions of cubic feet of natural gas were wasted. Many productive oilfields were not cared for properly and were thus permanently damaged. Improper drilling and production methods have caused wastes that are beyond accurate estimate in dollars and cents. There has been much waste in refining, and the utilization of petroleum and its products has been notably inefficient. With the growth of the industry, methods of producing, refining, and utilizing have improved, but the fact remains that no real and widespread effort to conserve petroleum and its products has been made until comparatively recently. Even now, a large part of the oil produced here is burned as fuel for steam-raising purposes and the like. Such utilization is inefficient and may be termed wasteful, for oil fuel should be burned in internal-combustion engines, and steam boilers should not be fired with a 3 per cent resource—petroleum, but with a 97 per cent resource—coal.

Table 1 and figure 1 give the domestic production of crude petroleum, the imports and the domestic consumption for the years 1914 to 1921. The table and figure show that during this period domestic consumption has increased rapidly—more rapidly than domestic production—that consumption has passed production, and that imports have increased enormously to make up the deficit.

TABLE 1—Production and consumption of petroleum in the United States during the years 1914-1921 inclusive.(a)

Year	Domestic production *b* millions of barrels	Estimated domestic consumption, millions of barrels *c*	Imports, millions of barrels *d*	Proportion of consumption produced in United States, per cent	Excess of consumption over domestic production, millions of barrels	Yearly increase of imports per cent
1914	266	261	17	102	—5	...
1915	281	273	18	103	—8	5
1916	301	319	21	94	18	13
1917	335	378	30	89	43	47
1918	356	413	38	86	57	25
1919*e*	378	418	53	91	40	40
1920*e*	443	531	106	83	88	101
1921*e*	470	526	125	89	56	18

a Table adapted from Summary of statistics of crude petroleum for January, 1921, and preceding months; issued by the U. S. Geological Survey.
b Marketed production.
c Sum of domestic production and net imports plus decrease of stocks or minus increase of stocks.
d Chiefly from Mexico.
e Preliminary figures of U. S. Geological Survey.

Although this situation may be temporarily reversed, and production may exceed consumption for a time, or there may be a temporary surplus from foreign fields, the petroleum situation should be regarded with respect to the years ahead rather than as a question of today. Any one who visions the future can see that the problem of furnishing enough oil to meet the inevitable demands is indeed of high importance.

The United States Geological Survey in February, 1922, estimated that there yet remained underground in this country 9,150,000,000 barrels of petroleum recoverable by current production methods. The country is now producing oil at a rate approaching 500,000,000 barrels a year, and at that rate the known reserves amount to less than 20 years supply. Even should the reserve be twice as large as estimated, our petroleum supply can not possibly be adequate for future needs.

CAUSES OF INCREASED CONSUMPTION.

Many factors have contributed to the enormously increased demand for petroleum during the past ten years. The greatest increase in demand has been for motor gasoline, caused by the rapid growth of the automotive industry. From 1909 to 1920 the number of cars and trucks in service increased about 3,700 per cent, whereas the production of gasoline increased from 13,000,000 to 1,162,500,000 barrels, or only 884 per cent. At the end of 1920 there were about 7,600,000 automobiles and 900,000 trucks in the United States, a gain of 450,000 and 500,000 respectively during the year. Air-planes and motor-boats are also increasing the demand for gasoline.

The demand for kerosene has increased in recent years, and its market price doubled in the two years 1919-1920, largely because of export demand and the increasing use in this country of

farm tractors, the majority of which burn kerosene or similar distillates.

Likewise the demand for gas-oil and distillate fuel-oil has increased. The demand for gas-oil to be used in enriching water gas is enormous and will continue, if it does not actually increase. Distillate fuel is used in certain metallurgical operations and this use will probably become larger. Gas-oil is also extensively used as raw material for the production of gasoline by cracking processes.

The growth of industrial activity in many countries, and the larger use of automobiles, trucks, and tractors, has greatly increased the demand for lubricants. In June, 1920, the production of lubricating oils in the United States reached the highest point on record—3,165,474 gallons a day—and refinery stocks of lubricating oils were more than 42,000,000 gallons smaller than in June, 1919. Exports of lubricating oils in 1920 were 48.1 per cent larger than in the preceding year.

The demand for residuum fuel-oil has grown rapidly during the past few years. Many coal consumers, including railroads and power plants, have turned to oil as fuel, because of its convenience. The tendency is particularly evident among ship operators. Our Navy uses large quantities of fuel-oil; the Shipping Board has stated that there will soon be over 1,700 oil-burning steamers under the American flag. The use of fuel-oil is not confined to shipping by any means; it is being used extensively on locomotives, in power plants and in industrial heating plants.

Evidently the demand for petroleum products is increasing; domestic production is falling behind domestic consumption; the peak of domestic production is in sight, and new sources of petroleum products, or substitutes for them must be found. The slump in consumption and exports in 1921-1922 is certainly not permanent and in no wise mitigates the seriousness of the petroleum supply problem.

POSSIBLE NEW SUPPLIES OF PETROLEUM.

IMPORTS.

The first possibility of new supplies of natural petroleum seems to lie in larger imports, chiefly from Mexico, the countries bordering the Carribbean Sea, and South America. Mexico has produced large amounts of oil, but there is considerable doubt as to its future production. Moreover much of the Mexican crude is heavy and asphaltic and is best suited for making fuel-oil and a little gasoline, some kerosene, and gas-oil containing too much sulphur for gas enrichment.

Other countries bordering the Gulf of Mexico and the Carribbean Sea are believed to contain oil deposits, but these are com-

paratively undeveloped and little is known as to the amount and quality of oil they will yield.

The United States, however, would be at a great disadvantage if wholly or even partly dependent on imports of a commodity so vital as petroleum. Mineral oils are essential to the Nation's safety, as was demonstrated during the World War, and to its commercial and industrial life, and every effort must be made to insure domestic sources of supply.

INCREASED PRODUCTION.

As noted on page 4 the United States has an underground supply of 9,150,000,000 barrels of petroleum recoverable by present production methods. Lewis[4] has estimated that ordinary production methods often do not recover more than 10 to 20 per cent of the oil underground. This waste suggests that new methods may be developed for lengthening the life and increasing the production of known fields. There is also the possibility that deeper drilling, at present restricted by high costs and inadequate equipment, may add to the supply by finding new productive sands in present fields. Moreover, it is probable that new fields of importance will be developed, but these expedients can hardly be expected to do more than postpone for a comparatively short period the time of critical deficiency. In brief, the mineral oils of the United States in the near future will evidently be supplied partly from domestic production (probably soon to decrease), partly from imports, and partly from substitutes, aided undoubtedly by more efficient methods of production and utilization.

INCREASED PRODUCTION OF GASOLINE AND MORE EFFICIENT UTILIZATION OF GASOLINE AND FUEL-OIL.

Much of the gasoline marketed in this country is "straight run", that is, distilled from the crude oil without being cracked, and without being blended with natural-gas gasoline. However, increasing quantities of cracked gasoline and gasoline recovered from natural gas are being satisfactorily blended with straight-run gasoline. Gasoline produced by cracking, or breaking down the molecules of heavier distillates, when properly produced and refined, is a satisfactory motor fuel. Natural-gas ("natural") gasoline is obtained by recovering the gasoline vapors in natural gas by compression or absorption. As it is usually too volatile or "wild" to use alone, it is ordinarily mixed or blended with heavier petroleum distillates before being marketed as motor fuel.

Discussing the efficient utilization of petroleum in connection with the motor fuel problem, Smith[5] states that it seems unlikely

4 Lewis, J. O., Methods of increasing the recovery from oil sands: Bureau of Mines Bull. 148, 1917, pp. 25-28.

5 Smith, N. A. C., The motor gasoline situation: Bureau of Mines Reports of Investigations, Serial No. 2084, February, 1920, 5 pp.

that the quality of gasoline produced can be increased by using more of the crude and making the product less volatile as has been done in the past. This is because the next fraction, kerosene, is in great demand, and its price is increasing. If any appreciable quantity of the lighter fractions of kerosene is included in the gasoline, it will cause a shortage of kerosene, with a consequent increase in price. The extraction of gasoline from natural gas is approaching its maximum since the gases that can yield gasoline have largely been exploited. Moreover the changing of fuel-oil (the supply of which is not subject to the same limitations) into gasoline by cracking processes involves a considerable expense and waste of material. Therefore unless the margin in price between the two is considerable, cracking is not profitable. Since the demand for fuel-oil is increasing, it may not be profitable to crack more fuel-oil into gasoline unless the price of gasoline is also increased.

Increased use of kerosene as motor fuel can be accomplished only if automobile users are prepared to pay a price greater than can be afforded in the regular uses of kerosene, as the normal production of kerosene is not more than enough for normal demand for kerosene.

Smith also states that the use of fuel-oil in the internal combustion motor may be the solution of the problem. If engineers are able to design an automobile or truck engine that will use fuel-oil in a satisfactory manner, they will relieve the danger of impending shortage and make possible the desirable extension of the use of the automobile and truck. An automotive engine using fuel-oil can compete against any other user of fuel-oil because this will be the most efficient use to which the oil can be put. Consequently, the supply of motor fuel would be limited only by the total supply of fuel-oil.

One important exception can be made to this statement, and that is, that part of the oil now used as fuel can be made into lubricants. The demand for lubricants has not been sufficient to consume all that might be made from the crude oils of the country, but it is certain to increase. After all, it must be emphasized that lubricants are the most necessary of all the products of crude oil. The industrial development of today would be impossible without lubricating oil and these lubricants can be obtained in sufficient quantity, so far as we now know, only from our petroleum.

The more extensive use of the Diesel type engine, replacing power installations that now burn fuel-oil directly, will make possible a much more efficient utilization of our petroleum.

SUBSTITUTES FOR PETROLEUM.

ELECTRIC POWER.

On the sea, the only fuel that can be substituted for oil is coal; on land, however, electricity can and undoubtedly will replace fuel-oil in many places. Some railroads,—The Chicago, Milwaukee and St. Paul, for example, with nearly 800 miles of main line electrified—have electrified parts of their main lines; others, including one of the largest users of fuel-oil in the country, are said to be planning extensive electrification. Various industries will undoubtedly make increasing use of electricity as the water-power resources of the country are developed. The proposed super-power system for the North Atlantic States includes both hydro-electric plants and huge steam-driven generating stations, some of which will be close to coal mines.

BY-PRODUCTS FROM COAL.

When bituminous coal, cannel coal, lignite, and peat are destructively distilled, tars of various kinds are produced, from which different products can be obtained. At present, gasoline blended with commercial benzol (recovered at by-product coke-ovens) and blends of gasoline, alcohol, and benzol are being marketed as satisfactory motor fuels. The heavier coal tar is being used extensively as fuel-oil in some sections of the eastern United States. As by-product coke-ovens replace the wasteful beehive ovens, more and more products from coal can be expected as substitutes for those obtained from petroleum, but coal by-products can be only of minor aid in supplying substitutes for petroleum. It has been estimated that if all the coal mined in the country for a year were coked in by-product ovens, the quantity of motor fuel that might be made available thereby would amount to only about 20 per cent of the annual domestic consumption of gasoline. The development of low temperature coal-carbonization processes may, however, greatly increase this potential yield.

ALCOHOL.

Unblended alcohol can be used as fuel in engines designed for higher compression than is ordinarily used with gasoline. Commercial alcohol can not be blended directly with gasoline, but mixtures of gasoline, alcohol, and benzol that will not separate on standing are being used in a limited way at present, and this use of alcohol will probably grow if the price of motor gasoline increases or the cost of making alcohol decreases. The calorific value of alcohol is lower than that of gasoline, but this is offset by its high efficiency. Alcohol can be produced from a large number of vegetable waste products, and probably can also be obtained in quantity from wood wastes, such as sawdust, although

this has not yet been demonstrated on a large scale. At present the production of alcohol is limited by the availability of raw material suitable for its manufacture. It is to be noted that land on which plants for the production of alcohol are grown, is no longer usable for the production of food.

SUBSTITUTE SOURCES OF LUBRICANTS.

It is difficult to see where satisfactory substitutes for petroleum lubricants can be found, except in shale-oil, a source of somewhat doubtful utility (page 123). The production of lubricating oils from petroleum has in no way reached its limit (p. 7), but the time is coming when substitute sources will be really needed. Good lubricating oils, such as castor oil, which is an excellent lubricant, can be made from vegetable sources, but the cost at present is very high.

SHALE-OIL.

As sources of substitutes for petroleum oils, the reserves of oil-shale in the United States stand out as most important. Oil-shales in Scotland, France, and Australia have been the source of products similar to those obtained from petroleum, and this fact makes it highly probable that, in the near future, oil-shales, which underlie great areas of this country, will be extensively worked.

The following pages treat of the history, technique, and economics of the oil-shale industry, and emphasize the value of our deposits. The technical and business problems that must be solved before these deposits are developed on a large scale, are not beyond our technical skill or financial ability. A broad view of the outlook justifies the statement that our oil-shale reserves insure a domestic supply of mineral oil products, similar to those obtained from petroleum, for a long time to come.

FOREIGN DEPOSITS OF OIL-SHALES.

SCOTLAND.

Oil-shale occurs in many parts of the world, but the shale deposits of Scotland, because of their having been the basis of a commmercial industry for many years, are the most widely known. These deposits lie mostly to the west of Edinburgh and south of the Firth of Forth.[6] The present operations of importance are all in one field in West and Mid-Lothian, the center of the field being about 12 miles west of Edinburgh. The field is rather well defined, stretching from the Firth of Forth west of the Forth Bridge, southward for about 16 miles between the Almond River and the Bathgate Hills in the region of Cobbinshaw and Tarbax. The oil-shales form part of the Calciferous Sandstone series at the base of the Carboniferous system.

The shales now being worked are black or brownish-black, break with a conchoidal fracture and give a dark brown streak. They are tough, flexible, free from grittiness and curl to the cut of a knife. The rich shale is very resistant to weathering, thus differing from the barren shales, called "blaes", because of their bluish gray color, by the miners. The specific gravity of the shales worked for oil, ranges from 1.71 to 2.30.

The shale is finely laminated, a property best noted in the spent shale, although the laminae in the raw shale can be seen distinctly under the microscope. Layers of rich and lean shale alternate, probably caused by alternate periods of rich vegetable growth followed by water overflow and sedimentation.

Good oil-shale resembles hard, dark wood, or even leather, and an experienced worker can determine its quality by the readiness with which it curls when cut with a sharp knife. This property and its color, weight and streak, enable it to be distinguished from the "blaes", although the latter often pass or grade into an oil-shale yielding enough oil and ammonia to be worked profitably.

Scotch shale operators distinguish plain and "curly" shale, the former being flat and smooth, and the latter contorted and polished or slickensided on the squeezed faces; the same shale may be partly plain and partly curly; the curly shales often, but by no means always, yield more oil than the plain shales. How-

6 Carruthers, R. G., The geology of the oil-shale fields: Mem. Geol. Survey Scotland, The oil-shale fields of the Lothians, 2d ed., 1912, part I, pp. 1-95.

ever, curly shales always yield well as only a reasonably rich shale curls under lateral pressure.

The shales worked at present (1921) yield about 25 U. S. gallons of oil and 36 pounds of ammonium sulphate to the short ton. Formerly shales yielding much more oil were mined, but they were worked out years ago. The shale-oil industry in Scotland started on a mineral yielding very little ammonia, but up to 130 gallons of oil to the ton. Some geologists do not consider this material, known as torbanite, from its occurrence at Torbane Hill, to be a true oil-shale (page 34). Since the exhaustion of the torbanite, the oil yield of the shales worked has grown smaller and smaller. As a rule the Scotch shales that yield the most oil yield comparatively little ammonia, and vice versa. Naturally the shales rich in oil were worked first, the ones now mined yield less oil but more ammonia.

American shales seem to differ from the Scotch in that the oil and ammonia yields increase together, that is, a good oil-yielding shale contains a comparatively high percentage of nitrogen. Another difference is that the oil yield of a given seam of American shale, below the zone of weathering, evidently does not decrease with increasing depth. See page 19.

In the Scotch deposits the beds that yield oil are inter-stratified with calcareous or siliceous beds, the "blaes" of the miners, that contain little or no bituminous matter. The geologist terms them "carbonaceous shales".

At different times some twenty different seams of oil-shale have been mined, but at present only about 10 are being worked. These range in thickness from 3½ feet to 8 or 10 feet, averaging about 5 feet. The seams are usually persistent, but of varying thickness; in places a bed of oil-shale grades into carbonaceous shale. The shale beds are folded, usually in small basins, and frequently are badly faulted. At Pentland, however, the seams extend for nearly two miles without serious faulting and dip uniformly. Most of the seams are now well mapped and correlated.

ENGLAND.

Oil-shale has been known for a long time in the Kimmeridge district of Dorset and has lately been found in and around Norfolk. This shale is commonly known as Kimmeridge oil-shale, from its occurrence in the Kimmeridge clays. It has been worked in a small way in Dorset from time to time for centuries. There are probably very large quantities of oil-shale in the district but thus far successful commercial development has been impossible, primarily because the crude oils from the shale contain from 6 to 9 per cent of sulphur and because of a low proportion of paraffin oils, are extremely difficult to refine. Proposals to use

the oil for fuel have amounted to little, as the present British Admirality specification for fuel-oil allows a maximum of not more than three per cent of sulphur, and efforts to reduce the sulphur content of Kimmeridge shale-oil to that amount have not been successful. Within the last two years, it has been reported that sulphur-free shales underlie those previously discovered in Norfolk, and a company has been formed to exploit them.

The yield of oil from core-drill samples of Kimmeridge shale varies from 15 to possibly over 42 gallons per ton, and sulphate of ammonia up to 30 pounds per ton.[7] The seams of shale that might be of economic importance seem to be rather thin.

WALES AND IRELAND.

Oil-shales of rather inferior quality were discovered in Wales a long time ago, and many years since considerable quantities were treated, but the competition of cheap foreign petroleum caused the abandonment of the industry.

In Ireland, fairly rich oil-shales are known, but the seams are badly disturbed and faulted, and are practically all overlain with sheet basalt.

CANADA.

Oil-shales occur in New Brunswick, Nova Scotia, Quebec, and Newfoundland. There are other deposits in other parts of the Dominion, but these have not been more than casually examined. The shales of the first two provinces mentioned are considered of importance and may be developed in the near future. A deposit of possible value has also been reported on the west coast of Newfoundland.

In 1907 a large quantity of shale from New Brunswick was sent to the Pumpherston Oil Co.. works in Scotland for testing. The results indicated that 24 to 48 gallons of oil, and in some cases, over 70 pounds of ammonium sulphate could be obtained per ton of shale.[8] In the Scotch retorts the throughput of the richer of these shales was much slower than that of Scotch shale.

AUSTRALIA.

Oil-shales occur in New South Wales and Queensland. In addition, a peculiar spore deposit, known as tasmanite, which yields considerable oil, occurs in Tasmania. According to recent reports, a large company has obtained an option to these tasmanite deposits and is to start the construction of retorts at once

7 Strahan, A., Special reports on the mineral resources of Great Britain, Vol. VII; Lignite, jet, Kimmeridge oil-shale, mineral oil, cannel coals, natural gas. Part I, England and Wales. Mem. Geol. Survey, 1918, pp. 29-30.

8 Ells, R. W., Joint report on the bituminous or oil-shales of New Brunswick and Nova Scotia, also on the oil-shale industry of Scotland: Canada Dept. Mines, Bull. 55, 1909. 57 pp.

(1921). Official reports of the Tasmanian Government estimate that the possible quantity of shale available is 12,000,000 long tons. The oil obtained is said to be of excellent quality.

Two grades of shale have been mined and retorted in New South Wales for many years; one termed "Kerosene shale", a rich variety, probably a torbanite, partly used for gas enrichment; and the other, "seconds", more like ordinary oil-shale, retorted locally. The two grades are identical in nature and origin, differing only in richness.

Much of the following description of the Australian oil-shales and their occurrence, is abstracted from articles describing the industry of New South Wales in detail.[9]

OCCURRENCES OF THE SHALES IN NEW SOUTH WALES.

Oil-shale occurs in New South Wales mostly in lenticular patches, rarely more than a mile long, but sometimes several miles wide, in the upper coal measures of Permo-Carboniferous age. The seams range in thickness from an inch or two to 4½ feet or more.

The most important earlier workings were at Hartley Vale, Joadja and Torbane, in the Capertee Valley. At Hartley Vale the shale was rich and 3½ to 4 feet thick, with about 10 inches of lower grade shale below that had to be removed with an axe. At Capertee there was 5½ feet of good shale with a few inches of splint above and below. The important works, at present, are mining the Capertee-Wolgan bed at Newnes, in the Wolgan Valley. This seam, the most extensive yet discovered in New South Wales, is 14 to 50 inches thick. On the Capertee side, one tunnel is said to have exposed for a distance of over 4,000 feet a seam averaging 3 to 4 feet thick.

Other shale deposits of lesser importance occur in the Blue Mountains and in the Hunter River valley.

NEW ZEALAND.

To work a so-called oil shale, which is probably a lignite of high sulphur content, a plant of the Scotch type was erected in 1900 at Orepuki. The work has since been discontinued on account of the low grade of the shale, the poor quality of the oil produced, and unexpectedly high operating costs. In 1901, 12,048 long tons of shale, valued at £6,024, were mined; in 1902 only 2,338 tons; and only a negligible quantity from that time to date. Besides the deposit at Orepuki other oil-shale deposits have been discovered in different parts of New Zealand, but that at Orepuki appears of most potential importance.

---

9 Steuart, D. R., The oil-shales of the Lothians, Part III, The chemistry of the oil-shales: Memoirs Geol. Survey, Scotland, 1912, p. 163.
Carne, J. E., The kerosene shale deposits of New South Wales: Dept. Mines and Agriculture, New South Wales, 1903, 333 pp.
Handbook of the mineral products of New South Wales: Dept. Mines, New South Wales, 1920, pp. 13-16.

AFRICA.

Oil-shale has been discovered in southern Rhodesia, the Transvaal and Natal, but as far as is known, no active development has been undertaken, although proposals have been made recently. Other deposits are reported in Morocco, Angola and—perhaps the largest of all—in the Belgian Congo. No reports giving the size of the deposits are available. As a rule the shale seams are relatively thin, and the oil yield low.

FRANCE.

The most important oil-shale deposits are at Buxières-les Mines and Saint Hilaire (Allier), which have been worked since 1858, and at Autun (Saone et Loire), where operations started in 1862. It is interesting to note here that the first oil-shales ever worked were those of Autun, products made from them by Selligue having been exhibited at the Paris exposition of 1839. Selligue can really be considered the father of the oil-shale industry, although operations in France were soon outstripped by those of Scotland. According to Redwood[10] the oil-shale industry of France dates from the year 1830.

Abraham states that the shales in France occurring at Autun and Buxieres are not true oil-shales, but consist of semi-liquid asphalt associated with shales.[10a]

Besides the two fields mentioned, shales have been worked in the Departments of the Var, Basses Alpes, and Puy du Dome. Little work has been done in these fields, but recent reports (1921) indicate that development of the shales of the Boson field in the Var is about to begin on a large scale.

In the Buxières region are three oil-shale works that obtain yields of oil varying from 13 to 26 gallons per ton. The specific gravity of the crude oil ranges from 0.850 to 0.900 and yields refined products as follows:[11]

Products obtained from crude shale oil at Buxières.

	Specific Gravity.	Per cent.
Lamp oils	0.810-0.820	28-30
Heavy oils	0.870-0.925	30-40
Pitch		29
Loss in refining		12-20

The heavy oils are said to make rather good lubricants, and from them is obtained a good grade of paraffin wax. The pitch finds a ready market for use in the manufacture of asphalt preparations. The average normal annual production of shale in the Allier district is about 67,000 tons.

10 Redwood, Boverton, Treatise on petroleum: Vol. 2, 1913, p. 87.
10a Abraham, Herbert, Asphalts and allied substances; 1920, p. 158.
11 Petroleum World, Oil industry in France and Alsace: Vol. 16, Sept. 1919, p. 372.

The Autun field, the most important of the French shale districts, has an area of about 250 square kilometers. Several beds of oil-yielding shale are inter-stratified with coal, gravel, clay, limestone, and conglomerates. The deepest bed mined yields about 12 gallons of oil per ton. A middle seam, called the "big seam" and the most important of those worked, yields 12 to 24 gallons per ton. Upper seams have been worked to a small extent. About 110,000 tons of shale are mined annually in the Autun field.

When refined the oils from Autun give products approximately as follows:[12]

Products obtained from crude shale-oil at Autun.

	Specific Gravity.	Per cent.
Lamp oil	0.820	35-40
Heavy oils	0.860-0.868	4
Green oil	0.895	25
Pitch	0.960	20
Loss in refining		14

According to Redwood[13] the shale-oil industry in France is not remunerative, due principally to the poor quality of the shale. He also states that the oils are very difficult to refine. However, the present heavy import duty on oil enables several deposits to be successfully worked.

JUGO-SLAVIA.

Large deposits of oil-shale have been known to exist near Alexinatz for many years.[14] Some of the shale was tested years ago in one of the Scotch works, and is said to have yielded over 45 gallons of oil to the ton. The main bed is said to be more than 90 feet thick; above and below it are beds of lower grade.

Recently a prospecting right covering an area of about 120 square kilometers has been granted to a newly-discovered area of oil-shale[15] at Radovagne, near Belgrade. The deposit contains two kinds of shale, a rich brownish-yellow shale, and a black bituminous shale. The former covers an area of 30 square kilometers, and is one meter thick at the outcrop. In laboratory tests the shale yields about 15.5 per cent of oil by weight.

SPAIN.

In the Pedro Martinez Basin, province of Granada, two mines have cut several beds of bituminous or oil-shale which range in thickness from less than one to nearly three meters, the total thickness thus far exposed at any one point being about 5 meters. The deposit has not been well explored, but it is believed

12 Petroleum World, reference cited.
13 Redwood, Boverton, Treatise on petroleum, 1913, Vol. 2, p. 90.
14 Redwood, Boverton, idem, Vol. 2, p. 91.
15 From unpublished report of H. I. Smith, U. S. Bureau of Mines.

to underlie an area of at least 200 hectares and to contain 9,800,000 cubic meters of shale, which on the average will yield 12.00 to 12.35 per cent of oil by weight. Other deposits of oil-yielding shale are known, especially at Rubielos de Mora (Castellon) and Mora (Teruel), but no data are available as to their extent and richness.

SWEDEN.

Oil-shales occur in various parts of Sweden, the principal one at Kinnekulle, Ostergotland.[16] These deposits are called locally "alum" shales, and are said to be extensive. Apparently they will yield a good percentage of oil and ammonia. In 1919 it was reported that development on a commercial scale would be undertaken, but later reports are that the plans have been abandoned.

BULGARIA.

There are said to be large deposits of oil-shales near Breznik, Radomir, Popovtzi, Kazanlik, and Sirbinovo.[17] In the deposit near Brezmk alone the surface shale is estimated at about 30,000,000 tons. The shale beds seem to be thick enough to be considered suitable for commercial working. Their yield of oil, as tested by various chemists, ranges from 7 to 21 per cent, 13 per cent being about an average. The oil is said to yield less gasoline and kerosene, but more lubricating oils and wax than the Scotch oil. Also, the nitrogen content of the Bulgarian shales is less than that of Scotch shales. Three concessions to work these shales are said to have been granted.

GERMANY.

Oil-shales are known to exist in the Rhine Provinces and in the States of Hesse, Wurtemberg, Baden and Bavaria. The Bavarian deposits, which yield about 16 gallons of oil per ton, are to be worked in the near future, according to recent reports. During the war the deposits at Messel, Hesse, were worked to some extent.

In Saxony an important industry has been established around the distillation of brown coal. The technique of the industry is much like that of the oil-shale industry, but the products, except paraffin wax, are dissimilar.[18]

ITALY.

A concession to work a bituminous shale deposit in the province of Messina, Sicily, was granted in 1915. The shale is said to be readily accessible and to yield from 7 to 9 per cent of a rather heavy oil containing 3.4 per cent sulphur.

---

16 Petroleum Review: The Swedish Oil Industry, Vol. 39, Dec. 28, 1918, p. 414.

17 Kemper, G. H., Deposits of oil-shale in Bulgaria: Commerce Reports, Bureau of Foreign and Domestic Commerce, April 17, 1920, pp. 540-541.

18 Scheithauer, W., Shale oils and tars and their products, 1911, 183 pp.

SWITZERLAND.

In 1915 a concession of about 8,000 acres was granted to work deposits of oil-sands and oil-yielding shales in the canton of Geneva. The shale is said to yield about 15 gallons of oil per ton, and the oil appears to be of good quality. The commercial possibilities of this deposit are not yet known.

ESTHONIA.

Oil-shale deposits extend from the west coast near Port Baltic, to the north shore of Peipus Lake. These shales have for some time been used as fuel. The deposits are covered with only about 6 feet of overburden and thus may be cheaply mined. It has been estimated that the deposit contains over 40,000 millions tons of oil-shale, in five seams, varying in thickness from 8 to 30 inches. One sample is stated to have yielded over 60 gallons of oil per ton. In 1921 production was confined to two small works, but reports indicate that development will soon begin on a larger scale. The Esthonian Government, in its efforts to prevent the formation of a monopoly, is not disposed to make terms attractive to the many companies which have requested concessions.

BRAZIL.

(Notes by D. E. Winchester)

Oil shales occur in Alagoas, Bahia, Maranhao, Sao Paulo and in south Brazil; the tertiary shales of Sao Paulo are by far the most important. **Alagoas**—Tertiary oil shales are exposed at low tide along the beach near Maceio, but appear to be of local interest only. Development was unsuccessfully attempted in a very crude way several years ago. **Bahia**—The "Turfa" at Marahu is of recent formation, local in extent and resembles peat rather than shale. It yields, on testing, more than a barrel of oil per ton. Development thirty years ago met with failure and the plant then installed is now in ruins. **Maranhao**—Thin, unimportant beds of Permian oil shale are known at several places. **Sao Paulo**—Thick beds of soft, easily cut shale yielding from 25 to 30 gallons of oil per ton, are within a few feet of the surface over a large area along the Rio Parahyba. A twenty-ton retorting plant recently installed at Taubate was idle during 1921. Considerable shale mined during the war was used in the cities of Rio de Janeiro, Sao Paulo, and Santos for gas production. **South Brazil**—Low grade Permian shale may be of considerable importance because of its great thickness (100 feet in places) and probable great extent. A retorting plant is reported to have been installed recently at Sao Gabriel.

OTHER COUNTRIES.

Oil-shale deposits of possible commercial importance are known in Argentina, Chili, and Uruguay. Because of lack of fuel resources in many parts of South America, some of these deposits may present excellent opportunities for successful development.

Discoveries of large deposits of oil-shale of good quality at Fushun, Manchuria, have been reported recently. Oil-shale has also been discovered in Mongolia. Deposits are likewise reported to occur in interior Arabia, and in the Machada Plain district of Syria. Little is known as to the amount and quality of these shales, or as to the exact locations of the deposits.

OIL-SHALES IN THE UNITED STATES.

Oil-shales are known to exist in many parts of the United States. Those to which particular attention is now being directed and which are, perhaps, of most economic importance, occur in the States of Colorado, Nevada, Utah, and Wyoming. The richest of these are in the Rocky Mountain region, and belong to the Green River formation of Eocene age. The shales of Nevada possibly are slightly younger than Green River, and most of those of Montana are certainly older (Upper Paleozoic). The black shales of the Eastern States: Illinois, Missouri, Indiana, New York, Kentucky, Ohio, Pennsylvania, and Tennessee, occur mainly at one general horizon in the Upper Devonian, although extensive deposits occur in the Lower Devonian and Ordovician; and those of California, which, strictly speaking, are not true oil-shales, possibly with minor exceptions, belong to the Miocene of Tertiary age.

OIL-SHALES OF THE ROCKY MOUNTAIN REGION.

The oil-shales of the Green River formation occur in Garfield, Mesa, and Rio Blanco counties of northwestern Colorado; in Uinta, Sweetwater, and Lincoln counties of southwestern Wyoming; and in Uinta, Duchesne, Carbon, and Wasatch counties of northeastern Utah. Of these areas, the largest now known is in Utah.

The Green River formation consists mostly (Frontispiece, Plate I), but not entirely, of shale. Some of the shale does not yield oil, and much of it is of too low grade or not in thick enough beds to be of present economic importance. According to Winchester[19] the Green River formation contains persistent beds up to 49 feet thick that will yield at least 35 gallons of crude oil to the ton. More thorough surveying and sampling may discover thicker seams of equal or greater richness. A recently discovered 20-foot seam, sampled across the face at one point, yielded over 60 gallons of oil to the ton, by laboratory test.

In Colorado, the Green River formation attains a maximum thickness of about 2,600 feet. The middle part of the formation contains most of the oil-shale, the upper and lower parts being practically barren. The oil-shales lie practically horizontal, except for occasional local displacements and foldings, and appear to be relatively uniform in thickness and yield. At least two definite workable beds yielding over 25 gallons of oil to the ton have been found practically wherever the formation has been carefully examined. In some places, above these two, there is a third bed of greater richness and usually of workable thickness. The rich oil-shale strata are from a few inches to over 30 feet

19 Winchester, D. E., personal communication, 1922.

A. OIL SHALE NEAR WATSON, UTAH

B. OIL SHALE NEAR GILLULY, UTAH

A. THIN BEDDED OIL-SHALE NEAR WATSON, UTAH

B MASSIVE CONTORTED OIL-SHALE NEAR DEBEQUE, COLO.

thick, and are inter-stratified with lean shales, barren shales, sandstone, limestones, conglomerates, and thin bedded oolites.[20]

The oil-yielding shales usually outcrop high on the sides of steep-walled canons (Plates I and IIIa), and are darker in color and more resistant to weathering than the barren beds. Their altitude above sea-level ranges from 6,000 to 8,000 feet. The regions in which the shales occur are mostly arid or semi-arid, sparsely populated, and in general not well supplied with transportation facilities.

Some beds, most of them too thin to be worked commercially, have yielded up to 90 gallons of oil to the ton in laboratory tests, and it is safe to assume that the Green River formation contains enough shale of workable thickness to supply a large amount of material that for many years will yield over 35 gallons to the ton. These shales will also yield upwards of 20 pounds of ammonium sulphate to the ton.

The oil-shales of the Green River formation occur in two principal forms, paper and massive (Plates IIa, b and IIIb), with many gradations. It is believed by many that the paper shale is merely a weathered form of the massive shale. Some workings seem to indicate this, as one tunnel driven in paper shale for more than 20 feet from the surface, found that this shale gradually became massive farther in. The massive shale has plain or curly form; the latter is more or less contorted and folded, and rarely shows slickensided surfaces. The paper shale is obviously foliaceous, but in much of the massive shale the foliated structure can be seen only in the spent shale, or in thin sections under the microscope.

The massive shale, which is black or dark brown, is exteremely tough and withstands weathering remarkably; it weathers to a bluish-white color on the surface, but weathering probably does not affect the oil yield of the shale to a depth of more than a few inches. The massive shale gives a brown, or brownish-yellow streak and has a conchoidal fracture. Fresh surfaces of many samples of the shale have a strong odor of petroleum which, however, quickly disappears. If fairly rich the shale can be ignited and burned.

The paper shale is gray to brown or even black in color, and its thin laminae are remarkably flexible, even when badly weathered, thus distinguishing it from ordinary carbonaceous shale. Weathering, as noted above, affects the paper shale much more than the massive.

The average specific gravity of the massive shale of this formation is about 2.00 (see page 156), the richer layers having

20 Winchester, D. E., Oil-shale in northwestern Colorado and adjacent areas: U. S. Geol. Survey, Bull. 641, 1917, pp. 139-198.

the lower specific gravity. Oil and ammonia yield seem to be directly proportional,—leaner beds containing a lower percentage of nitrogen. (See page 30.)

OIL-SHALES OF NEVADA.

The oil-shales of Nevada, which are probably of Green River age, occur mostly in the eastern district near Elko and Carlin. They differ physically and chemically from the shales of the Green River formation, and are usually somewhat lighter in color for the same yield of oil.

The Nevada shales generally lie in somewhat shallow basins of considerable extent. As a rule the beds are rather thin, dip steeply and are extensively folded and faulted. Mining conditions are not as favorable as in most parts of the Green River formation.

The richest seam of oil-shale in Nevada known to the writer is not more than 4 feet thick, yields in the laboratory 28.0 gallons of crude oil to the ton, and contains 0.43 per cent of nitrogen, equivalent to 40.5 pounds of ammonium sulphate to the ton. This bed dips at an angle of about 23 degrees. Above and below it are much thicker beds of leaner shales, probably of little economic importance.

OIL-SHALES OF CALIFORNIA.

The California deposits, for the most part, are hardly true oil-shales, as the greater part of the oil obtained from them occurs as such, and can be extracted by suitable solvents. The most extensive deposits in this State, which are part of the Monterey formation of Tertiary age, differ very much physically and chemically from the oil-shales of Scotland and from others in the United States. The mineral matter of the shale is diatomaceous, and the beds that yield oil occur in massive formation. (Plate IV.) They are much softer than the other oil-shales mentioned, not at all flexible, brown or brownish-yellow in color, and when freshly broken smell strongly of petroleum. Their oil yield varies greatly from place to place, but the average is not high; even the best do not yield much over 20 gallons to the ton, and their nitrogen content is very low. In many places the deposits are very thick, and are accessible; so notwithstanding their low yield of oil, they have commercial possibilities. Most geologists consider the Monterey shales to have been the origin of the oil in some of the oil-fields of California.

OIL-SHALES OF MONTANA.

The oil-shales of Montana occur at two distinct geologic horizons, one in the Phosphoria formation, probably of Permian

MONTEREY OR DIATOMACEOUS OIL-SHALE NEAR CASMALIA, CALIFORNIA

age, and the other in Tertiary beds.[21] The Phosphoria shales of western Montana are characterized by a rather high percentage of phosphates.

Near Dillon there are beds three feet or more thick that will probably yield up to 30 gallons of oil to the ton. The phosphate beds, associated with the shales, are possibly of future importance as a commercial source of phosphates.

The Phosphoria shales of this region are dark brown or black, give a brownish streak, and the richer portions are frequently oolitic (in rounded form or pebbles). On weathered surfaces the shales exhibit a great variety of colors, a bluish-white usually predominating. On fractured surfaces the shales are often slickensided. When freshly broken or rubbed they give off an odor of petroleum, and will burn fairly freely when placed in a fire. They carry a little pyrite disseminated in very small grains.

The richest of the shales of this locality will probably not yield more than 30 gallons of oil to the ton, and apparently there are no beds of workable thickness as rich as this. However, seams of workable thickness will yield up to 20 gallons, and contain as high as 0.77 per cent nitrogen (equivalent to 71 pounds of ammonium sulphate per ton), and about 2.0 per cent of phosphorus calculated as phosphoric pentoxide (P_2O_5). Beds associated with the oil-shale contain as high as 24 per cent of P_2O_5. There is no known method of treating the shales for their oil and phosphate content at the same time.

The shales of the Phosphoria formation outcrop along the slopes of the chief mountain ranges. In contrast with the shales of the Green River formation, the Phosphoria shales in general are in steeply dipping beds, and extensively faulted.

The Tertiary oil-shales of this district occur in long, narrow basins and are of small extent; they are inter-stratified with sandstones, sandy shales, and lignite. The shales occur at about the middle of the Tertiary beds, are light brown, on weathered surfaces nearly white, and break into thin, more or less flexible laminae on weathering. In contrast with the Phosphoria shales, they do not smell of petroleum when freshly broken, but will burn when exposed to a hot flame. Samples from some beds, up to 5 feet thick, have yielded up to 24 gallons of oil per ton on laboratory distillation, and contained from 0.1 to 0.90 per cent nitrogen. The beds rich in nitrogen seem to be poor in oil, like the Scotch shales.

---

21 Bowen, C. F., Phosphatic oil-shales near Dell and Dillon, Beaverhead County, Montana: U. S. Geol. Survey, Bull. No. 661, 1918, pp. 315-320.
Condit, D. D., Oil-shale in western Montana, southeastern Idaho and adjacent parts of Wyoming and Utah: U. S. Geol. Survey, Bull. No. 711B, 1919, 26 pp.

The Phosphoria formation also outcrops in southeastern Idaho and adjacent parts of Wyoming and Utah, where it carries black and brown shales that are practically barren of oil.

OIL-SHALES OF THE EASTERN UNITED STATES.

The black shales of the Devonian formation[22], capable of yielding possibly economic quantities of oil, are known to exist in the States of Indiana, Illinois, Kentucky, Ohio, New York, Pennsylvania, West Virginia, and Tennessee. Shales of the same formation, yielding notable quantities of oil, have been reported in Missouri, Kansas, and Oklahoma. Oil-shale is known to occur in Texas, also.

The black shales of the States mentioned, with the exception of Texas, belong generally to the Upper Devonian. Other extensive deposits occur in the Lower Devonian and in the Ordovician. In a few places oil-shales of Carboniferous age overlie coal beds worked by open pits, so that the removal of the overlying shale is necessary.

In most places the Devonian black shales yield oil, but the preliminary investigations of the United States Geological Survey indicate that the yields of oil and ammonia are so low that commercial development is usually not feasible. There is still the possibility that deposits of oil-shale of economic richness may be found in the States mentioned. Private individuals have made reports on shales—especially in Kentucky, Illinois, and Missouri—that are rich enough, because of the probable cheapness and ease of mining, to make their development almost if not quite as feasible as that of the shales of Colorado, Utah, and Wyoming. The Bureau of Mines has tested samples of Kentucky shale, said to be of workable thickness, that yielded 16 gallons of oil per ton and contained 0.62 per cent nitrogen, equal to a theoretical yield of 58.6 pounds of ammonium sulphate per ton.

The Devonian shales are black or brown, extremely tough, and usually contain a relatively high percentage of sulphur. On long exposure to weathering, the shale breaks into thin, fissile, ash-colored fragments which crumble and finally weather into a stiff clay.

THE BLACK DEVONIAN OR OIL-SHALES OF KENTUCKY.[23]

By far the greater part of the Devonian rocks, which with the Silurian, form about 3 to 4 per cent of the total area of the state, consists of Devonian black shale or oil-shale. This means that thousands of acres are amenable to steam-shovel mining. More-

22 Ashley, G. H., Oil resources of black shales of the Eastern United States: U. S. Geol. Survey, Bull. 641, 1917, pp. 311-324.

23 From notes prepared by C. S. Crouse, Professor of Metallurgy, University of Kentucky, Lexington, Kentucky, who secured geological data from W. R. Jillson, director of The Kentucky Geological Survey.

over much of the State is underlain at various depths with this material, which thus forms an immense potential source of oil, though it is practically valueless at present because underground mining would be necessary. These shales are known in different areas as Ohio shale, New Albany shale and Chattanooga shale.

The chemical composition of two samples of Devonian black shales is indicated in Table 2.

TABLE 2—Chemical analysis of black Devonian shales of Kentucky and Ohio.

	Madison County, Kentucky, per cent	Columbus, Ohio, per cent
Bituminous matter, water and loss on ignition	12.00	11.40
Silica (SiO_2)	63.12	60.35
Iron and Alumina ($Fe_2O_3+Al_2O_3$)	8.56	21.20
Phosphoric acid (H_3PO_4)	0.14	....
Calcium Oxide (CaO)	2.03	3.33*a*
Magnesium Oxide (Mg O)	6.26	2.95*a*
Potash (K_2O)	1.36	....
	93.47	99.23

a Calcium carbonate and magnesium carbonate respectively.

The main outcrop of Devonian shales forms a rough semicircle with a radius of about 50 miles, the city of Lexington being the center, and extends on the northeast and northwest to the Ohio River. There are numerous smaller outcrops in different parts of the State, and the major part of Kentucky, except the Blue Grass section, is underlain with shale at various depths; practically all of the oil wells scattered so widely throughout the State have shown the shale lying immediately over the oil sands. In fact gas is often found in the shales themselves.

The thickness of the shale exposed in knobs and hills differs with the amount of erosion, but ranges up to about 225 feet. Thus far since these beds have not been sampled systematically, positive statements on average oil yield are unwarranted, but in marked contrast to the western shales, the indications are that the yield is decidedly uniform not only throughout a single deposit but throughout those sections of the State that have been sampled.

On a fresh fracture the unweathered shales are velvety black, are fairly tough, and show small disseminated nodules of pyrite. A small splinter will burn in a candle flame with evolution of gas. The weathered shales are brown to black, softer, more easily broken, and are less uniform in oil yield. As they form a very small part of the total, their commercial possibilities may be disregarded.

A sample of the unweathered shale assayed by the Bureau of Mines yielded 16.5 gallons of oil (specific gravity 0.930) to the ton, and contained 0.40 per cent of nitrogen, indicating a theoretical yield of 37.8 pounds of ammonium sulphate to the ton. Table 26 (page 175) contains a distillation analysis, made by the bureau, of an oil from unweathered Indiana shale. This is similar to oil produced from Kentucky shale.

ECONOMIC IMPORTANCE OF THE BLACK SHALES OF THE EASTERN STATES.

Though the black oil-yielding shales of Kentucky have received more attention than those of neighboring states, the deposits in Indiana and Ohio deserve at least equal consideration. Nor should the shales of other Eastern States be ignored.

Indiana in particular contains very large deposits of these shales, which outcrop in the southeastern part of the State, between North Vernon and New Albany, and which are known locally as the New Albany shales. The Bureau of Mines and State of Indiana are now working in co-operation to establish the economic value of these deposits. The description given above of the Kentucky shales applies almost as well to the Indiana shales, though the average oil yield of the latter is apparently less than that of the Kentucky shales.

On first thought, the eastern shales do not appear very attractive, because of their low oil yield as compared with the western shales, but to offset this, to a considerable extent at least, the following factors must be considered in connection with the eastern deposits:

Possible method of mining. Thousands of acres of the shales can be cheaply mined by steam shovels, and as the beds are exposed to a great extent, in knobs and hills, gravity will be of great aid in mining.

Transportation. Large areas of shale lie directly on, or at very short distances from railroads, thus making the transportation of supplies to, and products from a plant, a very simple problem.

Market. The deposits are close to an unlimited, though now highly competitive market for products, and close to sources of mining and other supplies.

Water. It is generally conceded that a commercial shale-retorting plant should include a refinery. Water for condensing and for refinery use is plentiful through the shale district.

Properties of the shale. Experiments have shown that this shale will not coke on retorting and is easily crushed.

Properties of the oil. The oil produced from these shales apparently is of higher quality than that produced from many western shales, especially as regards amount and quality of motor fuel fraction.

Attention is also directed to the fact that a lean shale can probably be retorted more rapidly than a rich shale. Retort capacities seemingly are determined largely by richness of shale rather than units of shale treated. (See page 73.)

NATURE AND ORIGIN OF OIL-SHALE.

DEFINITION OF OIL-SHALE.

The writer defines oil-shale as follows: "Oil-shale is a compact, laminated rock of sedimentary origin, yielding over 33 per cent of ash and containing organic matter that yields oil when distilled but not appreciably when extracted with the ordinary solvents for petroleum".

According to Conacher[24] "oil-shales and torbanites form a group of materials which have in common the characteristic that on distillation they yield a product consisting typically of paraffin and olefins, and this feature is the source of their industrial importance".

Ashley[25] says that the line between a coal and a shale has never been sharply drawn, but he makes the suggestion "that material which, when burned, breaks down and yields an ash that goes through the grate bars and shows no tendency to maintain its original shape, is a coal, and that material which on burning yields an ash that tends to maintain its original shape, is a shale. The exact percentage of ash that should distinguish a coal from a shale can not yet be given, but until more exact figures are available, it is suggested that material that yields less than 33 per cent of ash be considered a coal".

Thiessen states[26] that "a shale is generally defined as a rock formed by the consolidation of clay, mud or silt, having a finely stratified, laminated or fissile structure. When such a rock contains organic matter it is termed carbonaceous or black shale; when the matter is of a bituminous nature it is called bituminous shale, and when rich in bituminous substances, yielding oil and gas on distillation, it is called oil-shale".

The definition given at the head of this section may be generally applied to all true oil-shales, but it excludes much material which has been considered of importance as a source of oil by distillation, such as the diatomaceous shales of the Monterey formation in California. A large part of the organic matter (probably oil as such), in these shales can be extracted with certain solvents

24 Conacher, H. R. J., A study of oil-shales and torbanites: Trans. Geol. Soc. Glasgow, Vol. 16, part 2, 1917, p. 164.

25 Ashley, G. H., Cannel coal in the United States: U. S. Geol. Survey, Bull. 659, 1918, p. 11.

26 Thiessen, Reinhardt, Origin and composition of certain oil-shales: Paper read before Chicago meeting of the Geological Society of America, Dec. 30, 1920, p. 1.

for petroleum. Practically all shales will yield small amounts of bitumens or hydrocarbons when extracted with various solvents. Table 3 below, indicates the solubilities of different shales.

In Table 3, the first column shows the source of the shale; the second column shows the amount of oil produced from the shale by distillation in the Bureau of Mines type of assay retort, expressed in gallons per ton; the third column shows the amount of oil recovered by such distillation, expressed as a percentage by weight of the raw shale; the columns under the heading, "Extraction with solvent", show first, the percentage (by weight) of the shale extracted by the particular solvent, and second, the relative amount of extract produced, expressed as a percentage of the oil yield by destructive distillation.

Similar results have been obtained with Scotch shales by Steuart.[26] This investigator found that Broxburn shale is soluble in various solvents as follows: Ether, 1.66 per cent; carbon bisulphide, 2.04 per cent; shale gasoline, 1.79 per cent, and a mixture of equal parts of shale gasoline and ether, 1.95 per cent.

The amount soluble, based on the weight of the shale, is small in all cases, but it is to be noted that a considerable percentage of the **organic matter** in the shale is soluble in most of the solvents used. A solubility of 2 per cent means an extraction of 40 pounds, or roughly, 5 gallons to the ton.

The extracts in no manner resemble the products of distillation, nor can they be considered oils as the word is generally understood, but without exception, the organic matter of all shales thus far examined is appreciably soluble in petroleum solvents. In other words, all these shales contain appreciable amounts of bitumen, though it is probably correct to say that they contain little or no oil as such. Oil-shales contain a substance, or substances, usually classed as a pyrobitumen, that by destructive distillation, or pyrolysis[27] yields oils somewhat similar to petroleum. These substances have been termed "kerogen" from two Greek words meaning producer of wax.

According to Steuart[28], Prof. Crum Brown suggested "the term 'kerogen' to express the carbonaceous matter in shale that gives rise to crude oil on distillation".

COMPOSITION OF OIL-SHALE.

What kerogen is, and how it formed, are subjects of much controversy. The various theories as to its origin are presented later (pp. 34 to 38). When by proper treatment it is sep-

26 Steuart, D. R., Oil-shales of the Lothians, Part III. The chemistry of the oil-shales: Mem. Geol. Survey Scotland, 2d ed., 1912, p. 159.
27 The word "pyrolysis" has been suggested by Mr. W. A. Hamor.
28 Steuart, D. R., work cited, p. 143.

TABLE 3—Solubilities of oil shales in various solvents for petroleum. [a]

Source of Shale	Oil yield by distillation: Gals. per ton	Oil yield by distillation: Per-cent-age	Extraction with solvent — By Carbon Tetrachloride: Per cent soluble	By Carbon Tetrachloride: Per cent of distillation yield	By Carbon Bisulphide: Per cent soluble	By Carbon Bisulphide: Per cent of distillation yield	By Acetone: Per cent soluble	By Acetone: Per cent of distillation yield
Kentucky	18.22	7.28	0.037	0.51	0.015	1.44	....	....
Soldier Summit, Utah	44.60	16.64	0.74	4.45	0.76	4.57	0.53	3.16
DeBeque, Colo.	35.75	13.24	2.04	15.42	1.85	13.97	1.33	10.04
Green River, Wyo.	58.65	22.67	1.195	5.27	1.27	5.58	1.22	5.35
Ione, Calif. [b]	52.00	19.65	7.555	33.46	5.83	29.68	10.98	55.80
			By Ether		By Benzol		By Chloroform	
Kentucky	18.22	7.28	....	....	0.06	0.82	0.14	1.99
Soldier Summit, Utah	44.60	16.64	0.745	4.48	0.91	5.47	1.05	6.32
DeBeque, Colo.	35.75	13.24	....	....	2.23	16.84	2.41	18.22
Green River, Wyo.	58.65	22.67	....	....	1.37	6.02	1.75	7.72
Ione, Calif. [b]	52.00	19.65	....	....	....	....	10.16	51.73

a Gavin, Martin J., and Aydelotte, John T., Solubility of oil-shales in solvents for petroleum: Bureau of Mines Reports of Investigations, Serial No. 2313, Jan., 1922.

b Considered a lignite by most investigators.

arated from the mineral part of the shale, it resembles a very old dry leaf mold. Under the microscope kerogen appears scattered through the ground mass of the shale as small globules or irregular streaks, ranging in color from yellow or reddish-yellow to dark brown or nearly black, and its abundance in a given bed is a measure of oil-yielding capacity. Kerogen is undoubtedly the source of oils obtained by retorting the shale.

Naturally, the mineral matter of oil-shales is largely clay, or aluminum silicate, and Scotch shales are composed of it almost entirely. Shales from different localities differ in composition of mineral matter, but the examples given in Table 4 may be taken as fairly representative. Attention is directed to the difference between the Scotch and the Green River shales, particularly as regards alkaline constituents.

TABLE 4—Analysis of ash of various representative oil-shales.

Shale	SiO_2 per cent	Al_2O_3 and Fe_2O_3 per cent	CaO per cent	MgO per cent	Total per cent	Remarks
Scotland						Sulphur
Good average *a*	55.60	34.77	1.55	trace	100.73*f*	0.94
Scotland-Broxburn, seam *b*	49.72	35.60	2.40	2.20	89.92	
Norfolk, Eng. *c*	49.5	30.5	11.7	1.2	92.9	
Australia, kerosene shale *d*	29.6	67.4	1.4	0.3	100.63*g*	
Casmalia, Calif., Monterey shale *e*	75.8	19.1	1.4	0.9	97.2	
Juab, Utah *e*	50.4	20.9	14.8	6.8	92.9	Bureau of
Elko, Nev. *e*	65.5	25.5	0.6	0.8	92.4	Mines No.
Clay City, Ky. *e*	52.0	19.1	12.5	8.2	91.8	P1006
DeBeque, Colo. *e*	48.8	17.9	16.6	8.6	91.9	P1010
DeBeque, Colo. *e*	47.3	18.2	19.3	8.4	93.2	P1011
Dragon, Utah *e*	45.8	16.4	23.9	7.9	94.0	P1012
Dragon, Utah *e*	46.8	17.5	23.9	8.0	96.2	P1013
Green River, Wyo. *e*	38.9	12.4	38.3	4.9	94.5	P1014
Green River, Wyo. *e*	41.9	18.8	17.6	10.9	89.2	P1015
Ione, Calif. *e*	43.0	38.1	8.7	2.5	92.3	P1047
Soldier Summit, Utah *e*	44.8	17.1	23.9	5.4	91.2	P1048

a Conacher, H. R. J., Personal communication.
b Steuart, D. R., The oil-shales of the Lothians, Part III. The chemistry of the oil-shales: Memoirs Geol. Survey, Scotland. 2d ed. 1912, p. 159.
c Forbes-Leslie, William. The Norfolk oil-shales: Jour. Inst. Petroleum Tech., Vol. 3, Dec. 1916, p. 22.
d Carne, J. E., Kerosene shale deposits of New South Wales: Mem. Geol. Survey, New South Wales, 1903, p. 272.
e T. B. Brighton, Bureau of Mines analyst.
f Includes 8.27 per cent soluble in water containing 0.92 per cent SO_3.
g Includes K_2O, 0.748 per cent, Na_2O, 0.322 per cent; phosphoric acid, 0.744 per cent. Total ash in raw shale, 16.12 per cent.

Table 5 gives the approximate analysis of various American and foreign shales. It should be noted that in many American shales, the carbonate percentages are so high as to make the volatile and fixed carbon determinations of doubtful accuracy.

TABLE 5—Proximate analyses of representative American and foreign oil-shales.

Shale	Moisture	Volatile	Fixed Carbon	Ash
Broxburn seam, Scotland *a*	7.50	16.5	5.2*b*	70.8
Dunnet seam, Scotland *a*	6.47	19.25	8.37*b*	65.91
Jubilee seam, Scotland *c*	1.92	22.41	1.81	73.86
Pumpherston No. 5 seam, Scotland *c*	1.70	21.35	5.93	71.02
Hartley, Australia, best export *d*	1.02	75.20	15.30	8.48
Hartley, Australia, retorting shale *d*	0.85	45.32	31.27	22.56
Elko, Nev. *e*	2.40	29.3	4.2*f*	64.4*f*
New Albany, Ind	0.85	15.86	5.54	77.78
Clay City, Ky. *e*	0.70	28.6	0.7*f*	70.0*f*
Soldier Summit, Utah *e*	0.80	38.10		61.1
DeBeque, Colo. *e*	0.10	45.70		54.2
DeBeque, Colo. *e*	0.10	42.30		57.6
Dragon, Utah *e*	1.10	42.20		56.7
Dragon, Utah *e*	1.05	34.35		64.6
Green River, Wyo. *e*	0.25	34.95		64.8
Green River, Wyo. *e*	0.15	48.05		51.8
Ione, Calif. *e*	8.50	62.90		28.6

a Steuart, D. R., The oil-shales of the Lothians, Part III—The Chemistry of the oil-shales: Mem. Geol. Survey, Scotland, 2d ed. 1912, p. 158.
b Includes sulphur.
c Carne, J. E., Kerosene shale deposits of New South Wales: Mem. Geol. Survey, New South Wales, 1903, p. 294.
d Steuart, D. R., work cited, p. 163.
e T. B. Brighton, Bureau of Mines, analyst.
f Ash and fixed carbon determination unreliable because of high percentage of carbonates in raw shale.

Nitrogen content is a measure of recoverable ammonia, and Table 6 and figure 2 show nitrogen content of several foreign and American shales. The almost linear relationship between oil yield and nitrogen content in the different shales of the Green River formation is best seen in figure 2.

TABLE 6—Nitrogen content of various representative oil-shales. a

Number on curve, figure 2	Shale	Nitrogen, per cent	Theoretical available $(NH_4)_2SO_4$ lbs. per ton	Oil yield, *b* gals. per ton
1.	Elko, Nev.	0.43	40.5	32.52
2.	Elko, Nev.	0.15	14.1	7.28
3.	Clay City, Ky.	0.62	58.5	18.22
4.	Soldier Summit, Utah	0.58	54.7	44.60
5.	DeBeque, Colo.	0.96	90.5	60.15
6.	DeBeque, Colo.	0.76	71.7	48.77
7.	Dragon, Utah	0.74	69.8	41.65
8.	Dragon, Utah	0.40	37.7	21.70
9.	Green River, Wyo.	0.35	33.0	23.45
10.	Green River, Wyo.	0.83	78.2	58.65
11.	Ione, Calif.	0.39	36.8	52.00

a Determined by Kjeldahl-Gunning Method. T. B. Brighton, analyst.
b Laboratory retort.

The nitrogen content of oil-shales ranges from a trace to over one per cent. In Scotland the nitrogen is the source of the most important by-product obtained from the shales—ammonium sulphate. The value of the nitrogen in the American shales is discussed on page 123. Apparently the nitrogen exists in combination with the kerogen or oil-yielding matter. Although, in Scotch oil-shale the nitrogen content seems to vary inversely

as the oil-yielding possibilities, in shales of the western part of the United States the reverse seems to hold, as is shown by Table 6. Apparently the eastern shales are like those of Scotland in this respect.

Oil-shales often contain small quantities of pyrite (iron disulphide, FeS_2), possibly pyrrhotite (Fe_6S_7 to $Fe_{11}S_{12}$) and gypsum (calcium sulphate, $CaSO_4.2H_2O$). These minerals probably contain most of the sulphur found in the shales, but some of it, as indicated on pages 43 and 47, is chemically combined in the organic matter of the shale. The large quantities of hydrogen sulphide given off at rather low temperatures are believed to be from this organic sulphur. Table 7 shows the percentages of sulphur in some shales of economic importance. In considering the commercial possibilities of a shale, its sulphur content and the sulphur content of the oil produced from it should be known, as high-sulphur oils are objectionable. Sulphur in oils causes bad odors, tends to make white distillates become discolored, and often prevents an oil from passing many of the present commercial tests.

Hydrogen sulphide is a toxic gas and it is produced in varying amounts when practically all oil-shales are retorted. The poisonous nature of this gas should be kept in mind by retort operators, so that workers may not be exposed to dangerous concentrations.

TABLE 7—Sulphur content of representative oil-shales.

Source of shale	Sulphur, per cent	Sample number
Scotland *a*	2.66	P1000
Elko, Nev. *a*	4.10	P1002
DeBeque, Colo. *a*	1.16	P1011
Ione, Calif. *a*	2.13	P1047
Soldier Summit, Utah *a*	2.16	P1048
Green River, Wyo. *a*	3.84	P1055
Dillon, Mont. *a*	2.98	P1058
Clay City, Ky. *a*	3.16	P1061
Watson, Utah *a*	0.48	P1065
Joadja, New South Wales (best grade) *b*	0.31	
Scotland, Roman Camp mine Jubilee seam *c*	0.35	

a T. B. Brighton, analyst.
b Carne, J. E., The kerosene shale deposits of New South Wales: Mem. Geol. Survey, New South Wales, 1903, p. 225.
c Steuart, D. R., The oil-shales of the Lothians, Part III, The chemistry of the oil-shales: Mem. Geol. Survey, Scotland, 1912, p. 154.

POTASH AND RARE METALS FROM OIL-SHALES.

Many oil-shales contain small quantities of potash, but in few cases even after complete retorting—that is, after removal of all volatile and fixed carbon—is the amount of water-soluble potash enough to make recovery feasible. It is not believed that oil-shales can be worked commercially for their potash.

Much discussion has been aroused by reports that various investigators have obtained gold, silver, and platinum from oil-shales, usually by secret processes. Assays made by the Bureau

of Mines on samples of oil-shale said to carry one ounce and more of gold per ton, with corresponding silver and platinum, have indicated that although gold was present, it existed only as traces of the order of 0.01 ounce per ton, worth about 20 cents per ton. No silver or platinum have been detected. As many of those who claim to have found gold and other precious metals in paying quantities state that the metals cannot be detected or recovered unless they are "unlocked" by preliminary treatment, the Bureau made a series of tests and assays to determine the validity of these claims. Several of the tests were made in the presence of, and according to the methods said to be necessary by one of the private investigators who has received much publicity from his reputed discovery of methods of recovering precious metals from oil-shales.

A shale from which this investigator claimed he had recovered \$7.50 in gold per ton was used in the tests. Assays were made on the raw shale, spent shale, decarbonized shale, decarbonized shale treated with chlorine at temperatures most favorable for the formation of gold chloride, and on shale decomposed by chromic acid. All the assay returns checked closely, and indicated that the shale carried less than 65c in gold. The fire assay return on the raw shale was the same as on the shale subjected to the various preliminary treatments.

This work and similar assays by the Bureau, as well as other investigators, indicate little chance of gold and similar precious metals being commercially recoverable from oil-shale. There is no particular reason why gold should occur in oil-shale any more than it does in sea water, and although it is possible that small partings or fissures in the shales—not the shale itself—might carry notable quantities of these metals, it is not believed that oil-shale will ever be a commercial source of gold, silver, platinum, or allied metals.

KEROGEN, THE OIL-YIELDING MATERIAL OF OIL-SHALES.

Little information is available as to the chemical composition of the organic oil-yielding material of oil-shales, the so-called kerogen. From a careful study of Scotch shales, Mills[29] gives the composition of an average sample as:

Ultimate analysis of Scotch shale.	Per cent
Carbon	25.27
Hydrogen	3.67
Oxygen	5.65
Nitrogen	1.14
Sulphur	0.49
Ash	63.78
	100.00

29 Mills, E. J., Destructive distillation: 4th ed., 1892, p. 50.

Undoubtedly part of the nitrogen and sulphur of the shale are in combination with the kerogen, but excluding these and the ash, he finds the chemical composition of kerogen to be as follows:

Composition of Kerogen.

	Per cent
Carbon	73.05
Hydrogen	10.62
Oxygen	16.33
	100.00

This corresponds to the empirical formula $C_6H_{10}O$. Mills therefore, believes that the kerogen molecule of Scotch shale can be represented by the formula $n(C_6H_{10}O)$, n being undoubtedly a large number.

Certainly the kerogen molecule, if kerogen is a definite chemical substance, is large and complicated. Although the kerogen in different shales shows certain resemblances under the microscope, it is not probable that the kerogen of all shales has the same composition, nor that the kerogen of any one shale is a definite chemical compound. The properties of the oil from different shales differ widely, even when the shales are retorted under identical conditions, as is shown by Table 8.

TABLE 8—Yield and nature of oil from different American oil-shales.

[Retorted under identical conditions in Bureau of Mines laboratory testing retort.]

Source of shale	Yield of oil, gal. per ton	Specific gravity of oil	Setting point of oil °C.	Sample No.
1. Elko, Nev. *a*	32.50	0.883	28.5	P1002
2. Elko, Nev. *a*	7.28	0.882	25.0	P1004
3. Clay City, Ky. *a*	18.22	0.924	below 15.0	P1006
4. DeBeque, Colo. *a*	60.15	0.899	25.7	P1010
5. DeBeque, Colo. *a*	48.77	0.906	25.0	P1011
6. Dragon, Utah *a*	41.65	0.904	25.7	P1012
7. Dragon, Utah *a*	21.70	0.897	27.0	P1013
8. Green River, Wyo. *a*	23.45	0.898	33.0	P1014
9. Green River, Wyo. *a*	58.65	0.899	32.0	P1015
10. Ione, Calif.	52.00	0.907	44.0	P1047
11. Soldier Summit, Utah *b*	44.60	0.881	31.0	P1048
12. Scotland *b*	17.64	0.865	34.5	P1000

a Average of duplicate closely agreeing assays.
b Average of several closely agreeing assays.

There is much conflicting evidence as to the origin of oil-shales, and evidently much research work must be done before the question can be definitely settled. In the following pages which present various theories of origin, it is well to keep in mind that all oil-shales may not have been formed in the same manner, and that different conclusions are reached by different investigators because different shales were examined. Likewise many of the theories may be correct in part, and many suggested methods of origin may have had something to do with the formation of oil-shale deposits.

Microscopic examination of oil-shales from all parts of the world indicates that they are composed of:

(1) Minute carbonized or partly carbonized frgaments of plants, often showing cellular structure, small spores, pollen and the like.

(2) Yellow or reddish-yellow, more or less spherical bodies, regarded as algae, spores, or globules of oil.

(3) Irregular streaks of reddish-yellow, dark-brown, or opaque material.

(4) Shells of small crustaceans, and parts of the skeletons and scales of fish.

(5) Mineral matter, such as sand grains and pyrite crystals.

There is little evidence to indicate that animal life had much to do with the formation of the oil-yielding material of shales, and much evidence to the contrary. Possibly the animal remains are the source of much of the nitrogen and sulphur.

In general the following opinion prevails: That the yellowish globules and yellowish or brown, often nearly black, streaks in the shales, are the sources of oil when the shales are distilled, and that they are probably of vegetable origin, or are petroleum as such or in some altered form. The fact that little real oil can be extracted from shales by ordinary solvents of petroleum seems to preclude the idea that the globules are petroleum as such.

Under the microscope, some shales show practically no organic matter, except the irregular globular yellowish bodies; others contain few globular bodies, the organic matter being in irregular dark-colored streaks. The first shale worked in Scotland, torbanite, is an example of the former; many of the shales of the Green River formation in the United States are examples of the latter. All degrees of gradations exist between the two extremes. Some investigators differentiate the origin of torbanite from that of oil-shale, but the consensus of opinion is that the origin is the same.

It is pretty generally agreed that the mineral matter of the shales was laid down in fresh-water swamps or lagoons, but opinions on the origin of the oil-yielding substance differ. Most investigators have examined shales such as those of Scotland in which the yellow globular bodies predominate, and less information has been published on the origin of the irregular dark colored streaks and globules prevalent in American shales.

Bertrand and Renault[30] believe that the torbanites were composed of accumulations of microscopic gelatinous algae, which were preserved from complete decomposition by an antiseptic solution, possibly derived from their own decay, and that the

30 Bertrand, C. Eg., and Renault, B., Pila Bibractensis et le boghead d'Autun: Bull. Soc. Hist. Nat. Autun, vol. 5, 1892, p. 159.

antiseptic solution is now represented by the opaque organic matter of the shales.

Jeffery[31] attempts to disprove the algal theory, and suggests that the supposed algae are deeply sculptured coats of the spores of vascular cryptogams.

Seward[32] believes that the so-called organic bodies might after all be found to be of inorganic origin.

David White[33] originally accepted the algal theory of Bertrand and Renault, but later transferred his support to the spore theory of Jeffery[34].

Cadell[35] suggests that the animal matter (entomostraca and fish remains) of the shales may be the source of much of the nitrogenous matter, and that shales now devoid of fossils, but rich in oil and nitrogen, may have derived their oil-yielding material from organisms with soft bodies and no shell. Later[36] he suggested that oil-shales are derived from an extremely macerated peat.

Steuart[37] concludes, from his own work and that of previous investigations that "oil-shales may therefore be composed of (1) vegetable matter which has been made into a pulp by maceration in water and preserved by combining with salts in solution * * *; (2) richer materials of many kinds, such as spores, which nature has provided with means for protection against decay, and (3) a proportion of animal matter." He also concludes that oil-shale "may be considered a torbanite containing a larger proportion of earthy matter from its original deposition, or is simply a torbanite which has deteriorated with age." He gives this reason for the variations in the organic matter: "In accounting for the differences between different shales and between shale, torbanite, etc., we must remember that during the deposition of the sediments, as the lapse of time was very great, the climate must have varied to some extent, and the algae and plankton generally of the lagoon must have changed also, together with the shore vegetation, producing pollen, spores and seeds. The differences and varieties might have arisen from changes in the proportion of, or nature of, the humus, the spores and the animal matter. And there are also the modifications produced by age, and the different conditions to which they have been subjected during their long history in the earth, such as warmth from depth or from proximity to intruded igneous rock."

31 Jeffery, E. C., On the composition and qualities of coal: Econ. Geol., vol. 9, 1914, p. 735. The mode of origin of coal: Jour. Geol., vol. 23, 1915, p. 220.

32 Seward, A. C., Fossil plants: vol. 1, 1898, p. 178.

33 White, David, Zinc and lead deposits of the Upper Mississippi Valley: U. S. Geol. Survey, Bull. 294, 1906, p. 26. The effect of oxygen on coal: U. S. Geol. Survey, Bull. 382, 1909, p. 50.

34 White, David, and Thiessen, R., The origin of coal: Bull. 38, Bureau of Mines, 1913; p. 3.

35 Cadell, H. M., The oil-shale fields of the Lothians: Trans. Inst. Min. Eng., vol. 22, 1901, pp. 29 and 314-71.

36 Cadell, H. M., The story of the Forth: 1913, p. 8.

37 Steuart, D. R., The oil-shales of the Lothians, Part III, The chemistry of the oil-shales: Memoirs Geol. Surv., Scotland, 2d. ed., 1912, pp. 164-5.

Scheithauer[38] believes that a "large number of marine animals perished on certain occasions", possibly as the result of volcanic eruptions, and states as evidence that the Scotch shale deposits have been shown to belong to the volcanic region.

Robertson[39] gives as his conclusions that "there is little resinous matter in oil-shales, and that the organic matter is a decomposition product of vegetable matter, of a nature like that found in peat and cannel coal", and produced by a definite combination of external conditions.

Davis[40] writes as follows regarding the microscopic studies he was making at the time of his death: "It is clear that the structureless material of the Green River shales probably originated in a collection of plant debris which has, by decomposition and the activities of bacteria and other microscopic organisms, passed into a jelly-like phase, which is to be found in certain kinds of peat deposits. The plant remains that have been found characterizing the shales from every locality from which they have been examined (Green River formation), are those of microscopic algae mixed in small percentage with pollen and similar parts of higher plants. Animal remains have been very rare in the material studied, and those noted were chiefly those of insects in a very fragmental state.

"It seems apparent, therefore, that the study of the microscopic structure of these shales as seen in vertical and horizontal sections, leads to the conclusion that the material was laid down originally in water, and that it passed through a series of stages of decomposition before consolidation and lithification had taken place. The remarkably well preserved state of the delicate plant structures which have been examined indicates very slight disturbances of the original material, and an almost entire lack of changes produced by the action of metamorphosing agencies since lithification."

Cunningham-Craig[41] advances the theory that an "oil-shale field may be considered as the relics of a former oil-field". That is, oil has been formed in argillaceous deposits from vegetal debris, has migrated into porous beds, and "if any argillaceous beds of sufficient absorptive capacity exist in the vicinity of oil rocks, they will gradually absorb and adsorb the heavier and unsaturated hydrocarbons, and by their smaller porosity, especially if capped by impervious beds, will protect the adsorbed liquid from displacement by lixiviation, while inspissation is gradually modifying the petroleum in the direction of kerogen." Craig also believes that torbanite, etc., differs from oil-shale in origin, and states that the

38 Scheithauer, W., Shale-oils and tars and their products: 1913, p. 12.

39 Robertson, J. B., A chemical examination of the organic matter in oil shales: Proc. Roy, Soc. Edinburgh, vol. 34, 1914, pp. 190-201.

40 Winchester, D. E., Oil-shale in northwestern Colorado and adjacent areas: U. S. Geol. Survey, Bull. 641, 1917, pp. 163-5.

41 Cunningham-Craig, E. H., Kerogen and kerogen shales: Jour. Inst. Pet. Tech., vol. 2, 1916, pp. 238-269. A treatise on British mineral oil: 1919, pp. 3-63.

former may be considered "an incipient form of petroleum developed, adsorbed and inspissated **in situ.**"

Conacher[42] believes, after exhaustive microscopic investigations, that the "oil-yielding material consists of resin fragments (yellowish in color and in shape usually like flattened spheres) which owe their external form either to their site of origin in the plant, or to attrition in transport." He believes[43] that petroleum has played no part in the origin of Scotch oil-shales or torbanites, and that the opaque organic matter of the shales contains the nitrogen, and represents the liquid putrefaction products of vegetable matter; further, that this matter yields the tarry products of distillation, with possibly some of the paraffins. He states that on distillation those shales containing the higher percentages of what he terms resin (the yellowish bodies) yield a larger quantity of lighter oil than those in which opaque matter predominates. "It seems that this opaque matter is of the nature of the amorphous groundmass of ordinary coal, and that from it is derived by distillation such products as are characteristic of coal tars (even low-temperature tars) while the yellow bodies are the source of typical shale products."[44] Further, he states, "evidently it is this material (the opaque matter) that yields * * * possibly the bulk of the nitrogenous compounds (including ammonia and the nitrogen combined with hydrocarbons.)"

With reference to the oil-shales of the Green River formation in the United States, Conacher[45] informs the writer that though they contain yellow particles, these bodies are not like those in the Scotch oil-shales and torbanites.

Mr. A. C. Thomson, chief refinery engineer of Scottish Oils, Ltd., has kindly supplied the writer with the following notes on an observation made by him in Brazil, which is of interest in that the actual formation of an oil-shale deposit is described:

"At a place named Taubatah, about 50 miles from Sao Paulo, a shale was worked for some time. In a search for outcrops about 5 miles from this place a flat strath extended for 2/3 mile on each side of the river; very marshy, with a strong vegetable growth (lichens) with small water pools throughout; muddy, fish were alive in some pools, and in others nearly dry, were dead in the mud. Knowing from exposures behind this level ground there were shale beds soft and damp, but well laminated, that lost 35 per cent of moisture by exposure in the sun for 48 hours, it was determined, for information concerning the formation of shales, to sink a well or pit. By means of a hastily improvised pump was successful in sinking to 20 feet from the surface; went through black mud and brown mud alternately. At 16 feet down, distinct lamination began, and between laminations a few well defined fish fossils were

42 Conacher, H. R. J., A study of oil-shales and torbanites: Trans. Geol. Soc. Glasgow, vol. 16, part 2, 1916-17, pp. 164-192.
43 Conacher, H. R. J., Same work, p. 185.
44 Conacher, H. R. J., Same work, p. 174.
45 Conacher, H. R. J., Personal communication, 1920.

found, showing exactly the same fish structure as those found in the river to-day. The black mud, or shales in formation, were put there when the vegetable growth was of long duration and the brown mud would form the brown earthy ribs always found in shale beds, which corresponds with the period of water overflow leaving a mud deposit over the vegetation ready for vegetable growth when the waters receded."

THE CHEMISTRY OF OIL-SHALE DISTILLATION.

To all intents and purposes, as has been shown, oil-shale contains no oil as such, the source of the oil being an organic substance termed kerogen. Although a tentative empirical formula for the kerogen of Scotch shale has been determined (see page 33), it is more than likely that kerogen is not a definite chemical compound, but is made up of molecules of various types and sizes, and that the oil-yielding materials or "kerogens" of different shales are unlike chemically.

Whatever its chemical composition, kerogen is undoubtedly of organic origin, and is made up of carbon, hydrogen, oxygen, with probably a little nitrogen and sulphur; also its molecules are undoubtedly large and complex. Kerogen, as such, is of no practical value, but by heating in a retort its complex and large molecules are "cracked" to form the more simple molecules of gaseous, liquid and solid hydrocarbons, and their derivatives, that are more or less similar to those derived from oil-well petroleums.

HYDROCARBONS.

A hydrocarbon is an organic compound of the elements hydrogen, H, and carbon, C. Generally speaking, the simplest and lightest hydrocarbon molecules form gases; the heavier and more complex molecules form liquids, and molecules still heavier and more complex form solids. As a rule, the specific gravities and boiling points of the hydrocarbons increase with the size and complexity of the component molecules.

For example, natural gas is chiefly the hydrocarbon methane, CH_4, sometimes with small amounts of ethane, C_2H_6, and higher hydrocarbons. Commercial gasoline, when made by ordinary distillation from a paraffin-base crude, is a mixture of several liquid hydrocarbons of the paraffin series, such as hexane, C_6H_{14}, heptane, C_7H_{16}, octane, C_8H_{18}, and others. The hydrocarbons of kerosene and the heavier fluid petroleum oils are composed of molecules still more complex and with more carbon and hydrogen atoms, such as tridecane, $C_{13}H_{28}$. The solid paraffins or waxes are generally considered to be composed of still heavier and more complex hydrocarbons, such as trikosane, $C_{23}H_{48}$, etc.

SATURATED AND UNSATURATED HYDROCARBONS.

Hydrocarbons of the paraffin series are said to be saturated, because the molecules are saturated with hydrogen; that is, they are unable to take up more hydrogen atoms. All the paraffin hydro-

carbons are members of the paraffin series. Their type formula is C_nH_{2n+2}, where n, theoretically, may be any number. (The hydrocarbon nhexacontane, $C_{60}H_{122}$ is the longest normal paraffin chain known.) The carbon atom is said to be quadrivalent or to have the power of combining with four other atoms, or groups of atoms, each atom or group having a combining power or valence of one. Graphically the valencies of the carbon atom may be represented

$$\begin{array}{c} | \\ -C- \\ | \end{array}$$

and thus the saturated methane molecule, CH_4, in which all these valencies are completely utilized, is represented:

$$\begin{array}{c} H \\ | \\ H-C-H \\ | \\ H \end{array}$$

A carbon atom may be combined with hydrogen atoms, the atoms of other elements and groups of atoms, and with other carbon atoms. Thus the ethane molecule, C_2H_6, has the graphic formula:

$$\begin{array}{ccccccc} & & H & & H & & \\ & & | & & | & & \\ H & - & C & - & C & - & H \\ & & | & & | & & \\ & & H & & H & & \end{array} \qquad \text{or } CH_3-CH_3$$

In each formula all the valencies of the two carbon atoms are completely utilized.

As the number of atoms in a paraffin hydrocarbon molecule increases, the structure of the molecule becomes more complex, and several molecules can have the same constitutional formula but different structural relations. For example, the hydrocarbon butane, C_4H_{10}, exists in two forms:

(1) $CH_3-CH_2-CH_2-CH_3$, the normal form (n. butane).

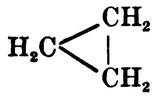
$$\text{or (2)} \quad CH_3-CH\begin{array}{l} \diagup CH_3 \\ \diagdown CH_3 \end{array}$$

the "isomeric" form (isobutane).

Butane and isobutane possess quite different properties. More-

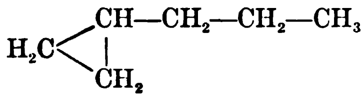
over, as hydrocarbon molecules become more complex, or larger, more isomeric forms can and do exist.

Saturated hydrocarbons of another series may be important constituents of shale oils. This is the saturated monocyclic or naphthene series, having the type formula C_nH_{2n}, but the structure of the molecules of this series is represented as a closed chain, or ring; for example, the first member of the series, the hydrocarbon trimethylene or cyclopropane, C_3H_6, is graphically indicated:



This hydrocarbon behaves much less like a saturated hydrocarbon than the cyclic compounds with five and six carbon atoms in the ring, probably because the valencies or bonds connecting the carbon atoms of cyclopropane are bent or strained from their normal position. The properties of cyclopentane, C_5H_{10}, and cyclohexane, C_6H_{12}, are very much like those of normal pentane, C_5H_{12}, and normal hexane, C_6H_{14},respectively.

Naphthene hydrocarbons may be complex; the formula above, by addition of a side chain may become:

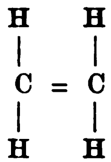


propyl trimethylene.

Saturated polycyclic hydrocarbons having the empirical formulae C_nH_{2n-2}, C_nH_{2n-4}, C_nH_{2n-6}, etc., may occur in shale oils. As the class name for this group indicates, the molecular structures are built up of more than one ring. Many lubricating oils are believed to be composed of polycyclic hydrocarbons.

A hydrocarbon molecule containing less hydrogen than the maximum possible is said to be **unsaturated**; if it is an "open-chain" molecule, it contains a smaller percentage of hydrogen than a saturated "open-chain" molecule of the same number of carbon atoms. Under proper conditions, unsaturated hydrocarbons can be saturated by direct addition of hydrogen atoms, whereas direct addition of such atoms to a saturated hydrocarbon is not possible. The three unsaturated series of hydrocarbons of possible importance in shale-oil are the olefin, diolefin, and acetylene series.

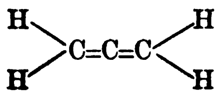
The type formula of the olefin series is C_nH_{2n}. The first member is the gas, ethylene, C_2H_4, sometimes graphically represented



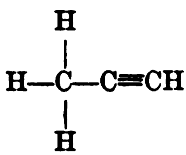
but usually $CH_2{=}CH_2$

In ethylene each carbon atom has an unsatisfied, strained, or uncombined valency or combining power, indicated by the double bond between the carbon atoms. The double bond does not represent a re-enforcement of a single bond, but indicates a point of molecular weakness or strain. Ethylene contains 14.3 per cent of hydrogen as compared with 20 per cent in the corresponding paraffin hydrocarbon, ethane, C_2H_6. The second member of the olefin series is propylene, C_3H_6, or $CH_2{=}CH{-}CH_3$. Among the higher members there are isomeric types. The olefins are probably the predominating unsaturated hydrocarbons in shale oils.

Another unsaturated series of hydrocarbons undoubtedly occurring in shale-oil is the diolefin; the first member of this series is an unstable gas, propadiene, or allene, C_3H_4, which contains 10 per cent of hydrogen and is represented by the graphic formula $CH_2{=}C{=}CH_2$, or



There is a series of compounds based on this hydrocarbon, with its more complex isomeric types, as in the other series mentioned. It may be noted that propadiene is isomeric with allylene, C_3H_4, the second member of the acetylene series, but the latter has the following structural or graphic formula:



or $CH_3{-}C{\equiv}CH$

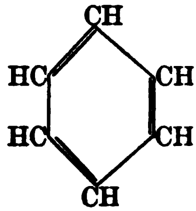
Another series of unsaturated hydrocarbons that may be present in shale-oils is the acetylene, though there is much experimental evidence that acetylenes are not present in oils produced by destructive distillation. The first member of the series is the gas acetylene, C_2H_2, which contains 7.7 per cent of hydrogen and is graphically represented:

$$H{-}C{\equiv}C{-}H \quad \text{or} \quad HC{\equiv}CH$$

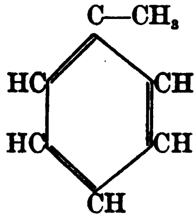
Each carbon atom thus has two unsatisfied combining powers or valencies, indicated by the triple bond. The type formula for

this series is C_nH_{2n-2}, which also serves as the type formula for the diolefin series.

Members of still another series of hydrocarbons—the aromatic or benzene series—probably are present in small quantities in shale-oil. The aromatic hydrocarbons are really unsaturated, but often are not so considered as many of their properties are like those of the saturated compounds. The type formula is C_nH_{2n-6}. The first member of the series is the liquid benzene, C_6H_6, graphically represented:



The next member is the liquid toluene, C_7H_8, or:



and so on.

As this brief discussion has indicated, hydrocarbon molecules can be indefinitely complex, and unsaturated and saturated groupings may be present in the same molecule. In addition, there are oxygen, nitrogen, and sulphur derivatives of hydrocarbons of all these series to be considered when shale-oil is studied.

THE NATURE OF THE DESTRUCTIVE DISTILLATION OF OIL-SHALE.

The oil-producing substance of oil-shales, though it is of variable composition in different shales, has been given the name kerogen, as before noted. Kerogen is only slightly soluble in the common organic solvents, such as carbon bisulphide, and is therefore classed as a pyrobitumen. Certain pyrobitumens[46]—those classed as asphaltic pyrobitumens—on being heated change into bitumens; that is, they become largely soluble in organic solvents, particularly in carbon bisulphide. Apparently the oil-producing materials of most oil-shales are correctly considered as asphaltic pyrobitumens, and thus, when an oil-shale is heated, the first step in the conversion of kerogen into oil and gas is the change of the insoluble pyrobitumen—kerogen—into a bitumen largely soluble in carbon bisulphide, and which, when extracted from the shale and freed from its solvent, is at ordinary temperatures a solid or semi-fluid, asphalt-like substance.[46]

The nature of the conversion of the pyrobitumen to bitumen is not known, but it is in accord with earlier deductions by Engler[46] that certain pyrobitumens, when heated to certain temperatures, become soluble in organic solvents. It has been suggested that the conversion may be a complete or partial depolymerization. It apparently begins at different temperatures in different shales, and ordinarily progresses rapidly at temperatures slightly over 400°C. If formed at this or a higher temperature the bitumen decomposes rapidly, producing gas, oil vapors, and fixed carbon, though if the temperature is not too high, and the vapors are removed rapidly, particularly with steam, some of the unaltered or only partly altered bitumen evidently may be carried out of the retort in the vapor stream. The change of the pyrobitumen—kerogen—into bitumen without appreciable decomposition of the latter, seemingly takes place at much lower temperatures if sufficient time is allowed.

---

46 E. E. Lyder and Ralph H. McKee first recognized that when an oil-shale is heated the pyrobitumen—kerogen—first changes into a bitumen and afterwards the bitumen decomposes, forming gas, oil, and carbon. This conversion has been studied by the Bureau of Mines and found to occur in all shales thus far examined. For a discussion of the nature of the conversion and a classification of various bitumens and pyrobitumens, the reader is referred to the following:

McKee, Ralph H., and Lyder, E. E., Thermal decomposition of oil-shales: Engineering and Scientific Paper No. 6, Columbia University, New York, August, 1921, pp. 6-10.

Abraham, Herbert, Asphalts and allied substances: D. Van Nostrand Co., New York, 1920. pp. 19-27, 56-58, 149-164.

Engler, C. Das erdöl: Bd. 1, 1917, p. 35.

Production of oil from oil-shale is thus broadly divisible into two stages, (1) the conversion of kerogen into a bitumen, and (2) the decomposition of the bitumen into gas, oil vapors and carbon. Some water may also be produced in the decomposition. In many cases the two steps of the process probably occur almost simultaneously.

Kerogen is undoubtedly composed of large and complex molecules, and the bitumen formed from it in the first step of the conversion must likewise be of large and complex molecular structure. In general, organic molecules are unstable under the action of heat in direct proportion to their complexity and size. A large and complex organic molecule, when heated to a high enough temperature, decomposes, breaks down, or "cracks" into smaller, simpler molecules. Thus the large and complex molecules of the first-formed bitumen, when heated in the absence of air, decompose into simpler forms. Heating an organic substance to a temperature sufficient to decompose its complex molecules into simpler molecules constitutes the process of destructive distillation, often termed pyrolysis. Essentially the retorting of oil-shale is the pyrolysis of a heavy and chemically complex bitumen.

The degree of decomposition or cracking of a molecule subjected to heat and pressure depends on its composition, the degree and duration of the heat and pressure, the material with which it is in contact, and on still other factors. The physical and chemical properties of the mineral matter of oil-shales have a considerable effect on the products obtained. By prolonged heating at high temperatures it is possible to decompose a hydrocarbon molecule into its constituent elements—hydrogen and carbon—a result not desired in oil-shale distillation. At different temperatures and pressures, and with different substances in contact with the molecules undergoing distillation, products of widely differing characteristics can be obtained, and the number of such products of different nature that can be obtained from a given molecule increases with the size and complexity of the molecule. As there are in the kerogen of oil-shales substances that probably contain extremely complex molecules, and as the molecular composition of the bitumen formed from the kerogen probably differs in different shales and with different conditions under which it is formed, it is evident that many kinds of hydrocarbons and hydrocarbon derivatives can be produced from an oil-shale, and that products having different properties may be made from different shales or from the same shale when treated under different conditions.

The oils produced when oil-shales are destructively distilled seem to be chiefly mixtures of members of the paraffin, olefin,

diolefin, and naphthene series and their derivatives with some of the aromatic and possibly some of the acetylene series. These have boiling points ranging from less than 50°C. to more than 400° C., and specific gravities from less than 0.700 to over 1.000. From this mixture of organic compounds comprising shale-oil, commercial products may be obtained by refining. In addition to the oils, permanent or fixed gases are produced when the shales are retorted. These gases are chiefly carbon monoxide, carbon dioxide, hydrogen, hydrogen sulphide, and the lower members of the various hydrocarbon series, those of the paraffin and olefin series predominating.

As organic molecules decompose in so many different ways and yield hydrocarbons and hydrocarbon derivatives of different series under different conditions, the commercial distillation of oil-shale can not be a haphazard process, but must be carefully controlled in accordance with conditions most suitable for obtaining the desired quality of crude oil. It is possible to distill under conditions that yield no oil but only gas and carbon as final products, but it has not yet been possible to produce oil alone without gas. Because the complex molecules break down in so many different ways under conditions that vary but slightly, and because in commercial practice mechanical and economic limitations render practically impossible the exact control of retorting conditions, it is not possible to produce from an oil-shale only the hydrocarbons desired. The process of distillation can not be stopped just when the required decomposition has taken place. Some of the hydrocarbons in the oils produced will be those desired, but others will be objectionable and must be removed by refining. A fact to be emphasized is that the properties of the crude shale-oil, and therefore of its products, are greatly influenced by the conditions under which the oil is produced. It is also evident that when an oil-shale is heated light distillates are not produced at low temperatures and heavier oils at the temperature increases. A heavy bitumen is first produced, and this decomposes into a mixture of vapors of light and heavy products, from which fractions may be separated by fractional condensation, or better, by bulk condensation and fractional distillation of the bulk condensate—the crude oil. (See page 179.)

It is possible that no conversion of pyrobitumen into bitumen, as previously described, takes place when certain shales containing oil-producing materials classed as non-asphaltic pyrobitumens are retorted. This does not invalidate the foregoing discussion. If bitumen is not first produced, the pyrobitumen, of large and complex molecular structure, decomposes directly into gas, oil vapors, and carbon; that is, there will be one less step in the process of conversion. The point emphasized here is that re-

gardless of the exact mechanism of the conversion of kerogen into oil, it is fundamentally the decomposition or cracking of heavy and complex organic molecules.

In the commercial distillation of oil-shale the main object is to obtain profitably the highest possible yield of the best oil. The oils having a so-called "paraffin base", those chiefly composed of members of the paraffin series of hydrocarbons, are most suitable for commercial production of refined oils, such as gasoline, kerosene, and the like. During destructive distillation organic molecules tend to loose hydrogen atoms. Shale-oils frequently contain more unsaturated than saturated hydrocarbons and are said to be highly unsaturated. In commercial petroleum products some of the olefin and practically all of the diolefin and acetylene hydrocarbons are objectionable; some give a bad odor to the oils; also, by oxidizing or slowly combining with themselves—a process known as polymerization—they form gums and resins. In particular, the more highly unsaturated compounds must be removed in refining in order to make satisfactory commercial products. The diolefins especially seem to be responsible for gum formation. For a detailed description of the various oil and gas products from oil-shale, the reader is referred to pages 77 and 86 to 90.

As the kerogen of oil-shales contains oxygen, nitrogen and sulphur in addition to carbon and hydrogen, shale-oils usually contain fairly large amounts of nitrogenous compounds, such as pyrrol (C_4H_5N), pyridine (C_5H_5N), quinoline (C_9H_7N), and their derivatives; traces of compounds of the phenol type, such as phenol (C_6H_5OH); undoubtedly sulphur-containing compounds, probably of the type $C_nH_{2n}S$, such as $C_{10}H_{20}S$; and probably naphthenic acids—oxygen derivatives of the naphthene hydrocarbons. The color and odor of shale-oil fractions are probably due to the presence of sulphur and nitrogen compounds and naphthenic acids. Some of these compounds, if abundant enough in the oils, might be of value as by-products, but most of them, with the possible exception of the nitrogenous compounds, seem to be present in such small proportions that they merely constitute objectionable impurities which must be removed in refining. Even the nitrogen compounds, which may possibly be present in amount sufficient to make their recovery worth while, must be removed from the refined products, as they are particularly evil-smelling and discolor the oils badly.

In retorting oil-shale, therefore, the effort should be to produce an oil containing a minimum of objectionable hydrocarbons and other organic compounds, and a maximum of those that are satisfactory. The shale and the vapors from it must not be excessively heated in the retort or elsewhere until all of the oil

has been distilled. In retorting a particular shale there will probably be found a temperature and a period of heating to that temperature which will give the best results. Other factors, such as the size of the pieces of shale, the thickness of the charge, the method of heat transfer, the duration of contact of the vapors with the heated zone, the presence of different gases or vapors in the retort, and the like, have a bearing on the quality and quantity of the products. Usually, of course, theoretically best conditions must be somewhat modified to meet commercial considerations of feasibility and cost, and vary according to the product chiefly desired. For instance, from years of experience the Scotch oil-shale operators have determined the conditions for producing crude oil with a high percentage of paraffin wax. If an oil containing a high percentage of gasoline is desired, retorting conditions may be different (see page 127). A fact to be emphasized is that different shales probably will require different conditions for best results, and the best conditions may have to be determined for each different type of shale, and changed with changes in the market for finished produces. (See also page 121.)

USE OF STEAM IN DESTRUCTIVE DISTILLATION.

In Scotch practice steam is let into the vertical retort at the bottom, where it comes in contact with the residual or spent shale that has been slowly heating after all the oil has been distilled from it in the upper part of the retort. When the shale reaches the bottom of the retort its temperature is such that the steam reacts with its fixed carbon, probably in accordance with the reaction:

$$C+2H_2O=CO_2+2H_2$$

This is probably the main reaction, but the temperature is high enough to cause some production of water-gas, a mixture of hydrogen and carbon monoxide, according to the following equation:

$$C+H_2O=CO+H_2$$

This reaction may be theoretical; indications are that the carbon monoxide forms by the action of carbon dioxide on carbon:

$$CO_2+C=2CO$$

It has long been known that much of the nitrogen of the shale remains in the spent shale as long as carbon is present, and is freed from the shale as the carbon is removed. A large part of the nitrogen seems to be fixed in relatively stable compounds, and can be recovered only by burning these compounds or subjecting them to chemical action. Thus, when the nitrogen-containing compound is broken down with steam at high temper-

atures, amino (NH_2), or imino (NH) groups, or the like, may be reduced by the hydrogen from the steam to form ammonia (NH_3). When carbon reacts with the steam, nitrogen may possibly be set free to combine at once with free hydrogen, as follows:

$$N_2+3H_2=2NH_3$$

but it is not likely that this reaction takes place appreciably, if at all. As soon as the ammonia forms it must be removed to a zone of lower temperature, as it would rapidly decompose if it remained for any considerable time in the highly heated lower part of the retort.

In the Scotch industry the production of ammonium compounds is extremely important, and although some ammonia may be produced from oil-shale without the use of steam, the maximum yield of ammonia is obtained by using steam in the retorts, and heating the shales to high temperatures after the oil has been driven off. The use of steam also gives a greater yield of combustible gases; makes heat transfer more efficient; cools the spent shale, and in certain retorts may improve the quality of the oil, as pointed out in detail in later sections (pages 181 to 183). In general, American shales contain nitrogen, and the use of steam in retorting will probably depend to a large extent on whether the nitrogen is to be recovered as ammonia, and whether American shales will yield satisfactory oils without the use of steam in commercial practice.

A satisfactory method of heating an oil-shale is the prime essential in obtaining a satisfactory production of oil, and one important feature of the use of steam in retorting is the manner in which it distributes heat throughout the charge in the retort, and recovers it from the spent shale. If ammonium compounds are desired from American shales, the retorts will have to be steamed, but in the opinion of the writer, even though ammonia production be neglected the use of steam in retorting has so many favorable aspects that it deserves careful consideration.

THE HISTORY OF THE OIL-SHALE INDUSTRY.

BEGINNING OF THE INDUSTRY.

The production of oil from bituminous materials was known at least as early as 1694 when Ele, Hancock, and Portlock distilled "oyle from a kind of stone" from Shropshire, England[47]. As early as 1761, oils were distilled from bituminous or oil-shales for medicinal purposes[48] and as early as 1815, a works in England produced oil and ammonia by the distillation of coal[49].

THE OIL-SHALE INDUSTRY IN FRANCE.

In 1830, Laurent[50] obtained paraffin by distilling oil-shale, but the French oil-shale industry really began in 1838 with the perfection of a process developed by Selligue[51]. In 1839, at the Paris industrial exposition, Selligue exhibited samples of shale-oil products, light oils, burning oils, heavy oils and paraffin. The early patents of Selligue and DeBuisson (English Patent No. 10,726, 1845), describe distilling apparatus, the use of steam in distilling, and the products and methods of refining.

The French industry grew in importance until 1864, when it was checked and set back by imports of cheap American oils, and it has never fully recovered. It received another blow in 1893, when the French Government cut in half the import duty on petroleum. Up to 1903, the retorts used in France were heated to comparatively low temperatures and steam was not used in them. The introduction of the Scotch type of retort increased the yield of oils from the shales more than ten per cent, and in recent years the industry has given indications of recovering some of its former importance, its development having been stimulated by the high price of imported petroleum oils during the World war and the years 1919 and 1920. In 1920 there were reports that new work on a large scale was about to be undertaken, particularly in the Boson fields in the Department of the Var. The present high import duty on petroleum in France is also encouraging the industry.

In 1893, in the Autun and Buxières districts, there were over 400 oil-shale retorts in operation in 96 plants[52], but none of these

47 English Patent No. 330, (1684). Ele, Phil. Trans: vol. 19, 1697, p. 544.
48 Lewis' Materia Medica.
49 Holmes, J. H. H., Treatise on the coal mines of Durham and Northumberland; 1816, p. 18.
50 Laurent, Aug., De l'huile des schistes bitumineux et de quelques produits qu'on en obtient: Compt. rend, t. 4, 1837, pp. 909-912.
51 Selligue, A. F., French Patent No. 9,467, Nov. 14, 1838, addition Mar. 27, 1839.
52 Chesneau, G., L'industrie des huiles des schiste en France et en Ecosse: Ann. mines, 9th ser.; t. 3, 1893, pp. 617-674.

were of Scotch design. In 1905, of a total of 292 retorts in operation in 74 plants, 232 were of Scotch design. Letters from Scotch operators affirm that at present practically all the retorts in operation in France are of the Scotch type. In the year 1904, the production of oil-shale in France was 184,030 tons, and the average yield was 20.07 U. S. gallons per ton, for a total yield of 3,693,783 gallons[53].

HISTORY OF THE OIL-SHALE INDUSTRY IN AUSTRALIA.[54]

At the beginning of the industry in 1865-66, shale was retorted at Kembla Valley and at Hartley Vale. In 1878 retorting began at the Joadja deposit, near Mittagong. For a number of years the industry was successful at Hartley and Joadja, but gradually the exportation of high-grade shale for gas enrichment took precedence over retorting. Finally the Joadja works closed though the Hartley works continued operations restricted to refining crude oil from retorts erected near Capertee by the same company. Paraffin was recovered and the oil was used for gas enrichment in the large cities of the State.

In 1908 the Commonwealth Oil Corporation took over the Hartley and Capertee works, and built a new plant at Newnes, which started operations in 1911. Operations ceased about a year later, as the original large capital was exhausted before satisfactory results were obtained. The Scotch retorts installed proved unsuitable for the higher grade shales of the Wolgan-Capertee seam, which yield 64 to 128 gallons of oil to the ton, compared to about 25 gallons from Scotch shales. Intumescence of the shale fractured the retorts and prevented the gradual descent of the spent material. Explosions were frequent. In June, 1914, this company was reorganized as John Fell & Co. New types of retorts were built on the site of the old ones, and these have successfully overcome the difficulties previously encountered.

METHODS OF TREATING THE SHALES AND PRODUCTS RECOVERED.

Retorting and refining methods for oil-shales and shale-oils in Australia have been fairly similar to those in Scotland. Both vertical and horizontal retorts have been used, the verticals until about 1910 or later, being somewhat like the Young and Beilby type. (See page 67.) A bench of modern Scotch-type retorts erected more recently was not wholly successful, as noted above.

53 Aron, A., L'industrie française des schistes bitumineux: Ann. mines, 10th ser.; t. 9: 1906, pp. 47-75.

54 Data for this section were obtained from Mr. Douglas A. Fell, of Sydney, Australia, and the following works:

Steuart, D. R., The oil-shales of the Lothians, Part III, The chemistry of the oil-shales: Mem. Geol. Survey Scotland, 2d ed., 1912, p. 163.

Carne, J. E., The kerosene shale deposits of New South Wales: Dept. Mines and Agriculture, New South Wales, 1903, 333 pp.

Handbook of the mineral products of New South Wales: Dept. Mines, New South Wales, 1920, pp. 13-16.

The yield of the richest Hartley shale in horizontal retorts without steam was 130 to 170 gallons of oil to the ton[55]. The oil was light colored, had a pleasant smell, and could be refined easily. Its specific gravity was 0.855, and its setting point 58° F. The richest Capertee shale, treated in the same manner, yielded 96 to 102 gallons of oil to the ton. This oil was considered somewhat inferior to that from the Hartley shale; its specific gravity was 0.866 and setting point 58° F. The horizontal retorts used were made of boiler-plate, and were 12 feet long, 6 feet wide and 2 feet 2 inches high. Three days were required to distill a charge. Hartley shale when withdrawn from the retort and cooled when half distilled, resembled stiff rubber in appearance and elasticity.

These rich shales were used for gas making, while the poorer shales above and below the rich seam were retorted, yielding when treated in vertical retorts, crude oils as indicated in Table 9.

TABLE 9—Yield of products from Australian shales.[56] *(Quantities calculated to tons of 2,000 pounds and to U. S. gallons.)*

	Hartley shale		Capertee shale	
Oil (gal. per ton)	48.2		51.5	
Specific gravity	0.890		0.900-0.905	
Setting point, °F.	70		72	
Refined products from crude:	per cent	sp. gr.	per cent	sp. gr.
Spirit	4.02	0.720-0.780	1.2	0.730-0.760
Burning oil	10.2	0.800	....	....
Gas oil	16.98	0.850-0.860	58.0	0.850
Blue oil	40.77	0.900	16.4	0.935
		Melting point		Melting point
Paraffin scale	4.41	....	4.1	122-3° F.
Coke	3.59	....	....	....
Loss in refining	20.03	....	20.3	....
Ammonium sulphate (lbs. per ton)	2.9	....	9.4	....

At Capertee, the splint was kept apart from the retorting shale, as the distillate produced from splint is a sticky resinous substance which clogs the condensers, and gives a persistent yellow color to the wax, making it impossible to refine to whiteness.

The Commonwealth Oil Corporation's plant consisted of a bench of 64 late-type Scotch retorts. Each retort had a capacity of 2 to 2½ tons of shale per day. This bears out the belief that the capacity of a Scotch retort must be rated in terms of oil production, rather than shale throughput (page 73).

In 1921 the plant now operated by John Fell & Co. consisted of 32 Fell Patent retorts. These are vertical and of original design. Each retort has a daily capacity of 6 to 7 tons of 80 imperial-gallon shale. The shale is broken by a gyratory crusher into pieces 5 by 2 inches or smaller before going into the retort hopper. The operation of the retorts differs little from that of

55 Steuart, D. R., work cited, p. 163.
56 Table derived from Steuart, D. R., work cited, p. 163.

present-day plants in Scotland. Enough permanent gas is formed to heat the retorts, making the use of gas-producers unnecessary. A continuous oil-distillation battery of stills is used for the crude, using three continuous horizontal boiler-type stills, working in series, and discharging from the last into a coking still. Acid is recovered from the refinery tars, and used in making ammonium sulphate. Distillations and treatments generally correspond with Scotch refining practice.

For the year ending September 30, 1920, this plant retorted 16,090 long tons of shale; the average production per long ton being 79.7 imperial gallons. The crude oil produced finished products about as follows:

	Per cent.
Benzine and spirits	5
Kerosene	20
Gas-oil	25
Fuel-oil	20
Wax	5
Refining loss and coke	25
	100

The yield of ammonium sulphate is about 19.5 pounds per ton. The oil-coke produced is sold for household use.

PRESENT STATUS OF THE OIL-SHALE INDUSTRY IN AUSTRALIA.

Financially, oil-shale operations in Australia have not been particularly successful so far, largely on account of extremely high labor costs and peculiar labor conditions. However, by reason of a bounty offered by the Federal Government for the production of mineral oils, operations have been stimulated, so there is a possibility that a commercial industry of considerable size will be developed in the future.

The payment of this bounty is provided for in the Shale-Oil Bounty Act, 1917. The period in which the bounty may be paid is four years, from September 1, 1917, to August 31, 1921. (This period has been extended.) It is paid on crude shale-oil, as prescribed by regulation, produced in Australia from mined kerosene shale, and the rate of bounty payable in each year to each producer of oil produced by him at each separate deposit as prescribed, is as follows:

	Pence
On each imperial gallon up to 3,500,000 gallons	2¼
On each imperial gallon exceeding 3,500,000 gallons, and not exceeding 5,000,000 gallons	2
On each imperial gallon exceeding 5,000,000 gallons, and not exceeding 8,000,000 gallons	1¾
On each additional imperial gallon	1½

The maximum amount that may be paid in any one year is £67,500.

The bounty paid by the Australian Commonwealth on crude shale-oil produced during the 12 months ending June 30, 1919, amounted to £26,406. It was earned by one firm operating in New South Wales[57].

Table 10 shows the production of oil-shale in New South Wales from 1865 to 1919:

TABLE 10—Production of oil-shale in New South Wales from 1865 to 1919. a

Year	Quantity, long tons	Year	Quantity, long tons
1865	570	1893	55,660
1866	2,770	1894	21,171
1867	4,079	1895	59,426
1868	16,952	1896	31,839
1869	7,500	1897	34,090
1870	8,580	1898	29,689
1871	14,700	1899	36,719
1872	11,040	1900	22,862
1873	17,850	1901	54,774
1874	12,100	1902	62,880
1875	6,197	1903	34,776
1876	15,998	1904	37,871
1877	18,963	1905	38,226
1878	23,371	1906	32,446
1879	32,519	1907	47,331
1880	19,201	1908	46,303
1881	27,894	1909	48,718
1882	48,065	1910*b*	68,239
1883	49,250	1911	75,104
1884	31,618	1912	86,018
1885	27,462	1913	16,983
1886	43,563	1914	50,049
1887	40,010	1915	15,474
1888	34,869	1916	17,425
1889	40,561	1917	31,661
1890	56,010	1918	32,395
1891	40,349	1919*c*	25,453
1892	74,179		

57 Cambage, R. H., Personal communication, 1920.
a Carne, J. E., Kerosene shale deposits of New South Wales: Dept. Mines and Agriculture, New South Wales, 1903, p. 286.
b Figures for years 1910 to 1918, both inclusive, from Handbook of the mineral products of New South Wales: Dept. Mines, New South Wales, 1920, p. 14.
c Figures for 1919 calculated from data furnished by R. M. Cambage.

HISTORY OF THE OIL-SHALE INDUSTRY IN SCOTLAND.

The oil-shale industry in Scotland apparently originated independently of that in France. In 1850[58] James Young, who with a partner, had been refining the oil obtained from a small seep in Derbyshire, England, conceived the idea that oil was a product of coal, and had been distilled from the latter by the action of subterranean heat. He began experimenting with the production of oil from coals, and finally produced from cannel coal an oil containing solid paraffin. In the course of his experiments a highly bituminous material, called "Boghead coal" or "Torbanehill material", or simply torbanite, was discovered. This yielded upwards of 135 gallons of oil to the ton. In 1850 a plant was erected by Young and two associates at Bathgate, Linlithgowshire, for the production of oil from Boghead coal, and was producing crude oil in 1851. Young's original patent[59] covering the process of the production of oil from bituminous mineral substances was the subject of much controversy and litigation, caused mostly by arguments as to whether Boghead coal was a coal or a shale. Finally it was decided that whatever the material actually was, the lessors had acquiesced in its being treated as within the terms of the lease, and that legally it was coal; the decision was purely on a point of law and scientific evidence was practically disregarded.

In 1862, the deposits of Boghead coal became exhausted, and since then the oils obtained in Scotland have been almost entirely produced from the oil-shales of the lower carboniferous series of Midlothian and Linlithgow. These shales were first worked in 1862 by Robert Bell at Broxburn.

The industry developed rapidly in Scotland, though many of the companies, organized to treat oil-shales, failed financially or combined with others in order to make success possible.

Since 1851, over 140 different individuals or companies have entered the oil-shale industry in Scotland. In 1871, there were 51 active companies, but of these the number in existence in 1910 was only six. In 1919, the four largest companies—Pumpherston Oil Co. Ltd., Broxburn Oil Co. Ltd., Young's Paraffin Light & Mineral Oil Co. Ltd., and the Oakbank Oil Co. Ltd., together with a smaller company, Jas. Ross & Co., Philipstown Oil Works Ltd., which produced crude oil only—consolidated as one company, Scottish Oils Ltd. Thus practically there is but one oil-shale company operating in Scotland at the present time, although the individual companies still maintain their identity.

58 Early history of the Scotch oil-shale industry, taken largely from Redwood, I. I., Mineral oils and their by-products, 1897, p. 33; et seq; late history from personal communications and interviews with Scotch operators.
59 English Patent No. 13292, Oct. 17, 1850.

The years following 1864 virtually paralyzed the Scotch oil-shale industry. That year marked the beginning of the import of cheap petroleum oils from America, and witnessed the failure of a great number of Scotch oil-shale companies and the consolidation of many of the remainder. Such consolidation, the development of more efficient and economical apparatus and methods of retorting the shales and refining the oils, and the strictest economies in mining, retorting, refining, and marketing, are undoubtedly the reasons why the industry has survived since, and in the face of the competition caused by the ever-growing petroleum industry.

The growth of the oil-shale industry in Scotland can be seen at a glance in Table 11, which shows the amount of shale mined in Scotland by years up to and including 1919. The yield of oil per ton of shale retorted has steadily decreased, and although complete official statistics for this in recent years are unavailable,

TABLE 11—Production of oil-shale in Scotland from 1873 to 1919. a

Year	Quantity, long tons	Year	Quantity, long tons	Year	Quantity, long tons
1873	524,095	1889	2,014,860	1905	2,496,785
1874	362,747	1890	2,212,250	1906	2,546,522
1875	437,774	1891	2,361,119	1907	2,690,028
1876	603,538	1892	2,089,937	1908	2,892,039
1877	801,701	1893	1,956,520	1909	2,967,057
1878	788,704	1894	1,986,385	1910	3,130,280
1879	783,748	1895	2,246,865	1911	3,116,803
1880	837,805	1896	2,419,525	1912	3,184,826
1881	958,255	1897	2,223,745	1913	3,280,143
1882	1,030,915	1898	2,137,993	1914	3,268,666
1883	1,167,943	1899	2,210,824	1915	2,998,652
1884	1,518,871	1900	2,282,221	1916	3,009,232
1885	1,770,413	1901	2,354,356	1917	3,117,658
1886	1,728,503	1902	2,107,534	1918	3,080,867
1887	1,411,378	1903	2,009,602	1919	2,758,555
1888	2,076,469	1904	2,333,062		

a Figures from 1873 to 1910 taken from The oil-shales of the Lothians, Part III. The chemistry of the oil-shales, Mem. Geol. Survey, Scotland, 1912, p. 139. Figures from 1910 to 1919 from H. R. J. Conacher, personal communication.

Table 12 gives some data for earlier years that are of interest. The explanation for this decrease of oil-yield per unit of shale mined is the fact that though the richer shales have been pretty well worked out, improvements in mining methods, and the

TABLE 12—Production of oil-shale, and oil and ammonium sulphate therefrom in Scotland for various years. a

Year	1886	1890	1900	1910	1918
Shale produced, long tons	1,728,503	2,212,250	2,282,221	3,130,280	3,080,867
Ammonium sulphate produced, long tons	18,080	24,730	37,267	59,113	58,311
Yield of Ammonium sulphate per long ton of shale lbs.	23.4	25.0	36.6	42.2	42.4
Yield of crude oil per long ton of shale treated, Imperial gallons *b*	31.2	34.7	28.4	25.0	23.0

a Based on data in Home Office Report: Mines and Quarries. Part III, London, 1910, p. 276; H. M. Alkali Inspector's annual reports, London; and from personal communications from H. R. J. Conacher.

b These figures are approximations, since no official data are available regarding crude oil production; years 1871, 1879, 1887, 1893 and 1918, respectively.

technique of surface operations—that is, handling, retorting and refining—have made the working of the thicker, more easily mined, but leaner shales, economically possible.

It might be said that the history of the Scotch oil-shale industry is the story of the development of the present apparatus and methods for retorting the shale, and to a lesser extent for refining the products.

In the early years, good profits were made by some of the companies, and there were recurring periods of prosperity. Generally, however, the financial status of the companies did not become satisfactory until about 1900. During the World War and in 1919, operating costs became higher, labor difficulties increased, and the production of oil from the shales now being worked became so low that the refineries could not be operated to capacity. Under these circumstances, the severe fall in prices in 1919 brought about the reorganization of the industry, as mentioned, and the formation of Scottish Oils Ltd., a subsidiary of the Anglo-Persian Oil Co. Ltd. It is reported that the unutilized capacity of the refineries will treat petroleum imported by the Anglo-Persian Company from the Persian fields.

At the time this manuscript is being written (March, 1922), the Scotch shale plants have been practically shut down for some time. This is believed to be due to the present economic depression and the British miners' strike. Very recent reports indicate that the plants are again beginning operations under more favorable economic and labor conditions.

THE SCOTCH OIL-SHALE INDUSTRY.[60]

The oil-shale industry has been developed to a greater extent in Scotland than in any other part of the world. The amount of capital invested in leases, mines, works, refineries, and the like in Scotland is in excess of $12,500,000. In the opinion of the writer, those who are engaged, or who contemplate investing or becoming engaged in the oil-shale industry in the United States, will make no mistake by studying, in detail, the technique of the Scotch industry. It has been in existence since 1850, and while its technique may not apply in its entirety to American shales and conditions, the experience of Scotch operators and their methods of solving difficulties in commercial operations can be of great value to our future oil-shale industry.

SAMPLING AND DETERMINING EXTENT OF OIL-SHALE DEPOSITS.

Once a new deposit or seam of oil-shale has been located and is to be worked, careful exploration and sampling are undertaken. The dip of the stratum at the surface is taken, although this may be of little value as the Scotch shale seams are usually badly folded and faulted. The next step is a careful and extensive sampling of the seam or seams by core-drilling, to learn the position of the seam, its extent, thickness, and often evidence as to folding and faulting. For this work, diamond core-drills are now used.

If evidence obtained by core-drilling is satisfactory, a trial shaft is sunk to the shale seam, in order to obtain absolute information as to its thickness and position, and to obtain from 50 to 100 tons of the shale for a large scale retorting test.[61] If the evidence is favorable, the seam is opened in the regular manner.

Companies starting oil-shale operations in the United States should thoroughly sample and measure their deposits with core-drills or otherwise, in order to determine the amount of shale available, its position, and its richness, before expensive plant installation is attempted. A little time and money spent in this kind of work may save much money, which might otherwise be spent in premature plant development, not based on accurate information as to the amount and value of the shale in the deposit.

---

60 This section, which deals with the technical development and present status of the Scotch oil-shale industry, has been reviewed, corrected, and revised by Messrs. James Bryson, H. R. J. Conacher, and A. C. Thomson, all officials of Scottish Oils, Ltd., Glasgow, Scotland.

61 Caldwell, W., The oil-shales of the Lothians, Part II, Methods of working the oil-shales: Mem. Geol. Survey Scotland, 2d Ed., 1912, p. 97.

MINING.[62]

In Scotland, oil-shales are almost invariably mined underground. Quarrying or open-pit operations are now rarely possible. The shale seams frequently dip at rather steep angles, and, as a rule, several seams overlie one another, and are often worked from one main entry. An entry or "mine" is sunk from the surface to the seam or seams, and this entry is often an incline sunk along one of the seams from the outcrop. In this event horizontal crosscuts are extended to the other seams, each being worked independently and in turn the crosscuts serving as means of communication and transit. In other cases, the shaft may be vertical and several overlying seams worked in turn from the one shaft. With modern equipment, it is considered cheaper to operate a shaft than an incline.

In most respects, mining oil-shale is similar to the mining of bituminous coal. Usually the roof has to be supported with timber props, and frequently the walls of the main galleries and the entries are bricked. Structural steel and reinforced concrete beams are coming into general use for permanent main gallery roof supports. Provision is made for drainage, ventilation, and haulage to main galleries. By legal regulation, all shale mines must have two independent entries. Coal-mining regulations are applied to shale workings, and are rigidly enforced.

Mining of the shale is usually done by the "Pillar and Stall" or "Stoop and Room" method, although the "Longwall" system has been used to some extent. In the former method, the galleries are first extended to the limits intended for them, and then the pillars, which until now have been left between galleries and are much larger than the latter, are removed, beginning at the outside and retreating towards the incline or shaft. As the pillars are removed, the roof is frequently supported by timbers, which are later removed and used again. The roof, as a rule, gradually sinks in behind the maximum removal of shale, or after the removal of the supporting timbers, if these are used, and this movement is ultimately transmitted to the surface of the ground. Where several overlying seams are mined from one main entry, the upper or No. 1 seam is sometimes first worked out, then No. 2, and so on; but frequently the reverse order is followed. An average of 25 per cent of the shale is left in the mine as small pillars, or as finely-divided refuse and shale of poor quality.

Shale produced at the working face is loaded into cars holding from 1 to 1.4 long tons. The cars are then moved by manual labor or by horses, or in inclined workings are conveyed to the main entry by various gravity systems, the loaded car pulling an

62 Much of the following description is based on the paper of W. Caldwell, Methods of working the oil-shales, Part II of The oil-shales of the Lothians: Mem. Geol. Survey Scotland, 2d Ed., 1912, pp. 95-135.

empty car up to the face. When the distance is great, electric haulage is used. In the main entry, the cars are usually hauled to the surface by means of endless, over-running steel cables, operated by electric motors on the surface, if the entry is an incline. When the entry is a shaft, a skip is used. Mine-cars are run onto the skip, hoisted, emptied in the building above the shaft, and returned to the gallery on the skip.

Electric power and lighting are generally used in the mines except at the most remote faces. Current is generated by a power plant at the shale-retorting works. The exhaust steam from this power plant is used in the retorting process.

Drilling is usually done with hand ratchet drills, similar to ships' augers, but in recent years electric rotary auger-drills have replaced the hand machines in some few cases. Holes are drilled at a good rate by either method. Gunpowder in paper cartridges is generally the explosive used in the shale mines. It is important that the quantity of fines produced be as small as possible, as this material, unless well distributed, tends to pack in the retort and hinders the proper passage of gases. On an average, about 0.72 pounds of powder are required per ton of shale mined.

A man working at the face produces about 4½ tons of shale per day of 8 hours. By "men working at the face" is meant the miner and his helpers. A miner always has one helper, and sometimes more.

COST OF MINING.

Present costs in the Scotch oil-shale industry are lower than they were during and in the two years following the World War, but they are yet much higher than before it. The process of adjustment is still going on, and is complicated by serious labor difficulties. For these reasons, and because costs in Scotland can not be expected to apply to American oil-shale operations and conditions, actual figures for that country are not given in this report. However, in 1919, mining operations accounted for 53 per cent of the total costs of producing marketable products from the shale.

Approximately, the cost of mining is similar to the average cost of mining coal in the British Isles. If anything, it is a little lower, largely on account of the average greater thickness of the shale seams now being mined.

In 1919 the Scotch shale miner was paid on either a minimum rate basis or on a straight tonnage basis. The rates per ton varied widely, and were based on the character and thickness of the seam, etc. Frequently the miner must do work essential to the development, drainage, or ventilation of the mine, but which yields only a small tonnage of shale. The miners' strike of 1921 will probably change the rate paid, the working hours, and the basis on which the pay is calculated.

The miners paid their helpers, who were also in 1919 paid a war-time bonus by the companies. The miner furnished his own powder, which was sold to him at cost by the companies. He also supplied his own lamp and oil; also his drills, and paid for dressing them. Coal proportioned to the power in the mine amounted to about 50 pounds per ton of shale. The cost of timber during and since the war has been a large item of cost in mining shale.

MINE VENTILATION AND GASES.

The mining laws of Great Britain compel forced or induced ventilation of shale as well as coal mines. Dangerous quantities of harmful gases are not commonly encountered in the shale workings in Scotland, but there is often enough to be hazardous, if proper ventilation is not effected. Both methane (marsh gas or fire damp) and carbon dioxide (choke damp or black damp) are met within the workings. In crosscutting, methane frequently escapes from fissures in the working face, especially where approaching the shale seam. Usually there is not sufficient gas in the workings to cause the miners to use closed lights and naked flames are almost invariably used in shale mining, but occasionally, especially in raises, closed lights must be used.[63]

According to Gray[64] the percentage of carbon dioxide and methane in air at the working-face ordinarily ranges from 0.29 to 0.09 and 0.02 to 0.06 per cent, respectively. Samples taken in the return airway of a Scotch shale mine indicated carbon dioxide 0.32 to 0.05, and methane, 0.09 per cent to none.

Ventilation is effectively provided for in the Scotch shale workings.[65] As each mine has two independent openings, one serves as an air inlet and the other as an outlet. Ventilating fans may be of the blower or exhaust type, depending on whether they are placed at the inlet or outlet shaft. Air is given a definite circulation throughout the workings, directed by doors, stoppings, or cloth brattices, the brattices being chemically treated to make them fireproof. In steep workings, just after blasting, the general circulation is sometimes temporarily insufficient to provide satisfactory ventilation in the raises. In such cases, hand-blowers, usually operated by a boy, often make up the deficiency. The blower is set a suitable distance from the working face, to which it supplies air through a wooden or iron pipe.

On account of the more simple system of the workings, the ventilation of longwall workings is cheaper than stoop-and-room workings.

63 Caldwell, W., The oil-shales of the Lothians, Part II, Methods of working the oil-shales: Mem. Geol. Survey Scotland, 2d Ed., 1912, p. 119.
64 Caldwell, W., work cited, p. 120.
65 Caldwell, W., The oil-shales of the Lothians, Part II, Methods of working the oil-shales: Mem. Geol. Survey Scotland, 2d Ed., 1912, pp. 123-126.

WATER IN MINE WORKINGS.

Only small quantities of water are encountered, on account of the impervious nature of the shales.[66] It is usually handled by pumps, driven by electric motors.

EXPLOSIBILITY OF SHALE DUST.

According to McLaren and Clark,[67] the dust of Scotch oil-shale in the mines retards explosions rather than exhibits a tendency to propagate them. Careful experiments were made on dust collected from the Broxburn and Pumpherston mines to determine if it were inflammable in the presence of a methane-gas explosion. These tests indicated conclusively that shale dust was non-explosive, and actually had a tendency to retard a gas explosion. However, it will not do to take for granted, in view of the non-explosiveness of Scotch shale dust, that dust in shale mines in the United States will also be non-explosive, nor that dangerous quantities of explosive gases will not be encountered. Indeed, the writer has witnessed experiments that lead him to doubt the non-explosibility of some shale dusts. It is recommended, therefore, until it is known that such precautions can be safely dispensed with, everything should be done to avoid explosions of gas and dust, as is done in coal workings. (See also pages 115 to 118.)

CRUSHING.

Cars carrying the shale are examined by an inspector at the mine mouth. He rejects unsuitable shale, after which the cars are weighed, and the weight credited to the miner who has filled them. Each car has attached to it some leather or metal tag, serving as an identification mark for the miner. After weighing, the cars are dumped directly into the crusher, or hauled by endless cable to this machine, depending on the distance of the mine from the retorts. Automatic tippers are usually employed for dumping the mine cars into bins above the breakers, but if the shale is carried in standard gage railway cars, a hydraulic ram is used.

The crushers consist of a pair of horizontal toothed-rolls which revolve slowly towards each other. They are of heavy cast iron, made up of hollow cylinders about 2 feet long and 3 feet in diameter, the total length of each roll being 9 feet. The clearance between the ends of the teeth is about one inch. The teeth are about 2 inches long when new, 1 inch square at the base, and set 4 or 5 inches apart each way. The maximum size of shale passing the rolls is about 5 by 5 by 12 inches, and the average of the larger pieces is about the size of an ordinary brick, but thinner. The capacity of a pair of rolls is between 480 and 780 short tons

66 Caldwell, W., work cited, p. 127.
67 Caldwell, W., work cited, p. 120.

a day. Fines are not usually screened out after the shale has passed the rolls, because if they are properly mixed, they can be satisfactorily treated in the retorts. The rolls rotate slowly, and are belt or gear driven by steam engines or electric motors. It should be noted that the shale is both broken and crushed by the rolls. From the breakers the shale is hauled in cars by endless cable or chain up an incline to the top of the retort benches, and there discharged by hand into the retort hoppers.

SCOTCH OIL-SHALE RETORTS.

THE DEVELOPMENT OF THE SCOTCH OIL-SHALE RETORTS.

As has been noted, one of the predominating factors in the success of the Scotch oil-shale industry has been the development of economical and efficient retorts for the production of oil, so a statement regarding the manner in which this development took place seems worth while; especially as the present status of oil-shale in the United States bears a striking resemblance to the early days of the industry in Scotland, when many retorts were tried, and abandoned, or improved, until the perfection of the present types in the years 1895-1902.

In considering retorts and retorting processes, it may be well to point out that the trend in development has been to devise apparatus and methods to produce, as cheaply as possible, the highest yields of the products for which there was the greatest demand, or which could be most readily marketed.

Up to the year 1865 it was apparently unsuspected that ammonia could be recovered from oil-shales, consequently up to that time all efforts were bent on producing higher yields of the best oils from the shales. The first retorts constructed by Young[68] were ordinary ⌓-shaped coal-gas retorts. These were charged and heated, and after the oil had been driven off, were discharged and re-charged. These horizontal retorts were of many different sizes and shapes, all attempting to utilize the heat efficiency. Their charging capacity varied from about 550 to 900 pounds, although a few had a capacity of over 2½ tons.

It was soon learned that control of the heat was difficult in such intermittently operated retorts, and to overcome this, one inventor devised a horizontal retort, heated to a high temperature at one end only. The shale was continually charged at one end from a hopper, conveyed through the retort by a helical screw, and when spent was discharged from the other end into a sealed water-lute. This type of retort, although adopted by Young's company, proved quite unsatisfactory, and was largely replaced by vertical retorts of cast-iron pipes 10 or 11 feet high, oval or oblong in cross-section, and tapering downward. Top dimensions

68 English Patent 13,292 (1850).

were 12 by 24 inches. Inasmuch as the rich shale then being retorted expanded somewhat during distillation, and also intumesced or fused together considerably, this retort frequently clogged, and its manipulation was difficult.

Young and Brash then devised[69] tapered cast-iron retorts of circular cross-section, set with the larger end down and the discharge water-sealed. A vertical helical screw, gear-driven from the top, was placed in the retort, and during the retorting operation the screw was kept slowly revolving. This apparatus largely overcame difficulties due to the expansion and sintering of the shale, and was successfully used for about eight years.

About this time, Young's company introduced a vertical retort said to have been suggested by Dr. Price.[70] This retort was of elliptical cross-section, and had a taper much greater than before used. The large part was placed downward, and the discharge was, as usual, into a water-seal. The retorts were charged from a hopper, and about 16 hours after the retort had been first charged and heated, a small quantity of spent shale was raked out of the water-seal, and an equal amount of fresh shale passed from the hopper into the retort. The hopper was recharged every three hours. Six or eight of these retorts were set in a common furnace, and it is to be noted that the vertical screw conveyers were no longer necessary.

This retort was developed in 1860, and it quickly became apparent that it was a great improvement over the horizontal retorts. Although various types of horizontals were used for many years, they were gradually superseded by the verticals, until today the verticals are the only type used. These increased the yield of oil from a given shale by over 10 per cent, and although the oil from the verticals had to be distilled before chemical treatment, which was not the case with oils from the horizontals, even then the cost of the "vertical" oil ready for chemical treatment was less than that from the horizontal retort at the same stage. The yield of naphtha from the "horizontal" oil was over twice as large as that from the vertical, but the yields of illuminating and lubricating oils were higher and the amount of paraffin obtained from the "vertical" oil was nearly 50 per cent more than that yielded by the "horizontal" oil. Inasmuch as paraffin wax was, and still is, the most valuable product obtained from shale-oil in Scotland, the advantage of the vertical retort is apparent.

In 1861, steam[71] was first used in retorting in Scotland, for the purpose of removing the oil vapors from the retorts as quickly as possible, to prevent undue decomposition. Steam in the vertical retorts not only increased the yield of oil upwards of 25 per

69 English Patent No. 625 (1866).
70 Redwood, Boverton. A treatise on petroleum: Vol. 2, 1913, p. 94.
71 Redwood, Boverton. A treatise on petroleum: Vol. 2, 1913, p. 96.

cent, but also produced an oil having a greater percentage of paraffin wax than when it was not used. Since 1861, steam has been generally used in vertical retorts. In addition to its effect on the oil yield, the use of steam was found to make heat transfer more effective and its distribution more uniform, thus making control of temperatures less difficult. In the case of horizontal retorts, the use of steam does not present such great advantages, because of the difficulty of securing effective distribution.

In the Price type of vertical retort, coal was used to furnish heat. About 500 pounds of coal was used per ton of shale retorted. The average life of these retorts was 4½ years, at the end of which time the bottoms were distorted and cracked so much that the whole retort had to be replaced.

The construction and operation of this type marked the first great advance in the development of the present Scotch retort. The continuous vertical retort with an elliptical cross-section was adopted in preference to the horizontal, and the use of steam in retorting was introduced.

In 1865, Robert Bell[72] of Broxburn, accidentally discovered that the water produced in retorting the shale was valuable as a fertilizer. Up to this time this water had been wasted and considered a necessary nuisance. Bell noticed that the growth of grass was extraordinarily luxuriant in a field into which the waste water had been turned. An investigation drew attention to the ammonia content of the water, and the development of methods for the production of ammonium sulphate followed. Incidentally, it may be mentioned that the production of ammonium sulphate has been the life-saver of the industry in Scotland, as the oil yield alone would have been insufficient to make it pay.

From 1860 to 1873, various forms of horizontal and vertical retorts were tried, some designed to operate continuously and others intermittently, but none of these are particularly noteworthy and the use of the vertical types increased. In 1873 Henderson[73] patented his first vertical retort, which marked a second great step in the development of the Scotch retort.

The features of the 1873 Henderson retort are, (1) the spent shale was used as a fuel for heating the retort; (2) discharge of the spent shale was greatly facilitated; (3) the distillation products passed out at the bottom of the retort, instead of the top as in previous practice; and (4) the fixed or uncondensable gases resulting from the shale distillation were, after being scrubbed to remove naphtha and ammonia, led back to the retort and burned as fuel. The retorts were vertical, of elliptical cross-section, and

72 Redwood, I. I., Mineral oils and their by-products: 1897, p. 169.
73 English Patent No. 1,327 (1873).

of cast iron, four being set in a common oven and heated by two furnaces. The shale was charged from cars at the top of the retort, and after 16 hours was discharged in a spent condition into one of the furnaces below, in which was also burned the retort gas, as noted above. Periodically the shale-ash was dropped from the furnace into a car, which took it to the dump. Operation of the retorts was intermittent, the retort being emptied completely before a fresh charge was added. Volatile products were removed from the retort in a downward direction, and passed from the retort through a vapor-line in the bottom. Steam, somewhat superheated, was passed into the top of the retort, which, on account of the design of the furnace, was the hottest, and the bottom the coolest, part of the retort.

There is a difference of opinion as to the real economic value of the combustion of the spent-shale for fuel purposes in the 1873 Henderson retort. Since the shale was discharged in a comparatively cool condition, it undoubtedly required considerable heat to bring it to, and keep it at, a combustion temperature. The spent shale from this retort contained considerable free carbon (9 to 14 per cent), as retort temperatures were insufficient to permit the action of the steam on the carbon to progress to any important extent. This fixed carbon was, of course, burned in the furnace, and probably supplied more heat than was needed to keep it burning. In any event, the burning of the spent shale and the fixed gases, as fuel, reduced the amount of coal required in retorting by about 50 per cent. On account of the ease of charging and discharging, the labor required was cut 33 per cent.

The yield of oil from the first Henderson was about the same as that from the older vertical retorts, but the total yield of finished oil products, especially wax, was notably greater in the former. The yield of ammonia from the Henderson was, however, only about half as much as from the old verticals. The life of these retorts, under proper care, was upwards of five years.

About 1880[74] Young and Beilby discovered that the recovery of ammonia could be greatly increased, if after the removal of the volatile matter from the shale, the latter was heated to a high temperature, and at the same time submitted to the action of superheated steam. It was soon determined that the yield of ammonia was increased as the carbon was removed from the shale by this action, and that the maximum amount of ammonia could be recovered only if all the carbon were so removed.

In line with these discoveries, Young and Beilby, working independently, constructed retorts designed to recover the oil from the upper part of the retort, where relatively low temperatures were maintained; ammonia was formed at the bottom of the retort where the temperature was high. The work of these men

74 Redwood, Boverton, A treatise on petroleum: Vol. 2, 1913, p. 96.

demonstrated the advantages of these discoveries, but in each case mechanical problems in retort design and construction caused the failure of the retorts. They then combined their ideas, and in 1882 patented the Young and Beilby, or "Pentland" composite retort[75], which was the first appearance of the modern Scotch retort. With minor modifications, this type of retort is still in use in Scotland.

THE YOUNG AND BEILBY RETORT (1882).

Plate V is a reproduction of a sectional model of a bench of Young and Beilby retorts. This retort is also known as the "Pentland" composite retort, as the single retort performed two definite operations, (1) distillation of the oil from the shale at relatively low temperatures, and (2) after the oil had been obtained, the production of ammonia from the nitrogen of the shale at a comparatively high temperature, with the aid of steam.

In operation, a hopper or "jumbo" **a** (Plate V), which was common to, and served four retorts, was filled with shale. The hopper had four lids for charging. The shale passed into the cast iron part of the retorts, **b**, which was of circular cross-section, and tapered slightly from top to bottom. In this part of the retort the oil was practically all distilled from the shale, at a temperature of about 900° F. The shale residue then passed into the fire brick part of the retort, **c**, (joined to iron part **b** with a fire clay joint, **k**), in which the shale reached a temperature of 1300° F. and was subjected to the action of superheated steam, entering the retorts at **e**. (In later retorts low-pressure steam without superheat was used.) In this part of the retort much of the nitrogen of the shale was converted into ammonia by the action of the steam; while at the same time and in the same reaction the bulk of the carbon in the residue was converted into water-gas. Discharge doors, **o**, were opened every six hours and spent shale was removed, corresponding in volume to 850 pounds of broken raw shale, which was charged into the retort following the removal of the spent shale. Gases and vapors resulting from the distillation were removed from the top of the hopper, **a**, after passing through the fresh shale therein, through vapor off-take, **f**, into vapor main, **g**, and thence into the condensers. Some type of exhauster was used in the vapor-line system to keep a slight suction in the retorts.

The passage of the distillation vapors through fresh shale in hopper, **a**, was expected partly to refine the oil and make the primary refining distillation unnecessary, but this arrangement did not accomplish what was expected of it.

Gas was used as a fuel, and consisted of the fixed shale distillation gases, supplemented by producer gas made from dross

75 English Patents 1,377 and 5,084 (1882).

coal. For each eight shale retorts there were two gas producers, which were so operated that the ammonia produced from the coal was recovered along with the ammonia from the shale. Gas supplied by pipes, **p**, was admitted into and burned in flues, **h**. Spent shale discharged from the retorts dropped into cars running on track, **t**, and was carried to the spent shale dump.

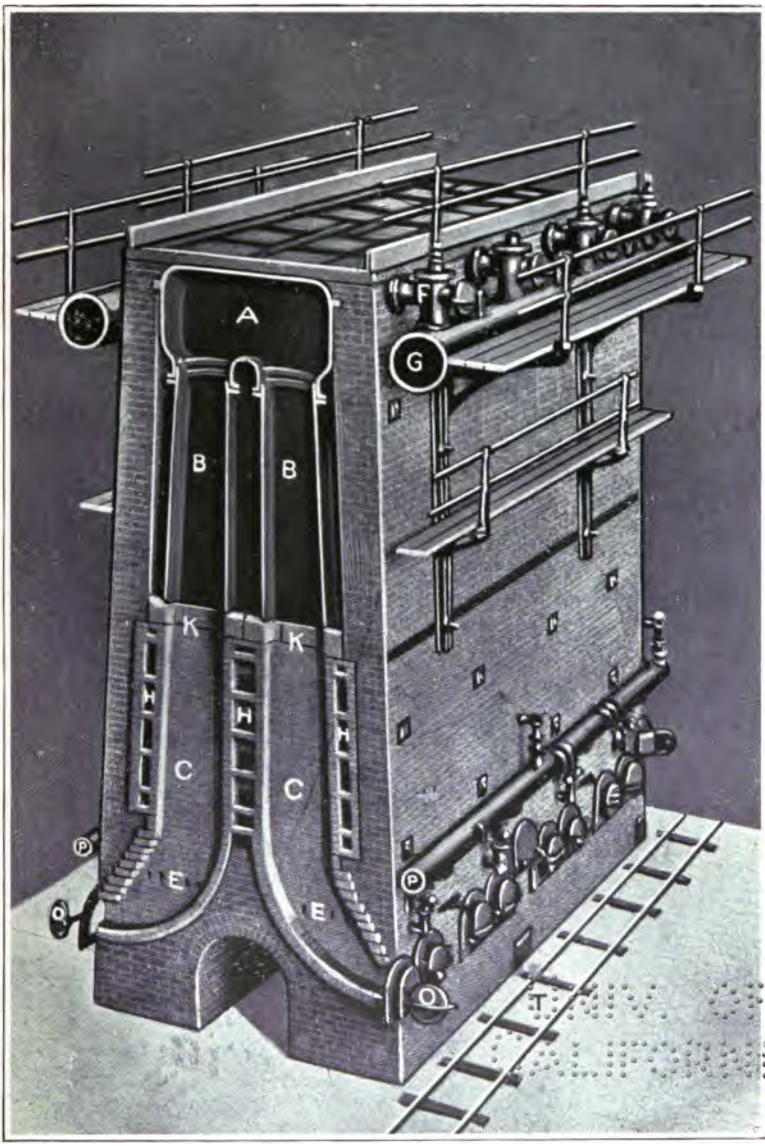
A bench of 80 retorts had a capacity of from 110 to 135 tons of shale per 24 hours, each retort thus putting through from 1.38 to 1.68 tons of shale per day. Each retort held about 3300 pounds of shale, so about 24 hours was required for a particle of shale to travel from top to bottom of the retort. Steam used varied with the quality of shale, but averaged about 108 gallons of water, as steam, per ton of shale. The steam was commonly the exhaust from the works power-plant, and quickly became superheated in passing through the hot spent shale in the bottom of the retort. The steam thus served several purposes: It recovered ammonia from the nitrogen of the shale; it made water-gas by combining with the fixed carbon of the shale; it cooled the spent shale, and in so doing transmitted heat back into the retort; it served to distribute the heat evenly in the retort; and finally it swept the distillation products from the retort, thus preventing their undue decomposition. Steam serves these functions in present-day Scotch oil-shale retorts. (See page 74.)

The Young and Beilby retort had a life of 6 or 7 years, if carefully operated. As in modern retorts, care had to be exercised in heating, especially to prevent the pulling apart of the joint between the iron and fire-brick parts of the retort. Coal consumption in this retort was from 100 to 300 pounds per ton of shale retorted. The oil produced was a little less in quantity but somewhat better in quality—especially in yield of paraffin and lubricating oil—than that produced in the old Henderson retort, but the yield of ammonia was from 2½ to 4 times as great as from the latter. The Young and Beilby retort, therefore, soon practically superseded all earlier types, until it was itself replaced by the more modern Young and Fyfe, Henderson, and Pumpherston or Bryson retorts. All these latter types are, however, based on the principle of the Young and Beilby, and essentially differ from the latter only in dimensions and in that they are continuous in operation instead of intermittent.

THE HENDERSON RETORT OF 1889.

In 1889, Henderson patented a new retort based on the Young and Beilby principle.[76] This retort in its improved form[77], which is shown in Plate VI, is used to the exclusion of others by the Broxburn Oil Co. Ltd., now a part of Scottish Oils Ltd.

76 English Patent 6.726 (1889).
77 English Patent 26,647 (1901).



(*Courtesy Mr. H. M. Cadell*)

YOUNG AND BEILBY'S "PENTLAND" RETORTS, 1881

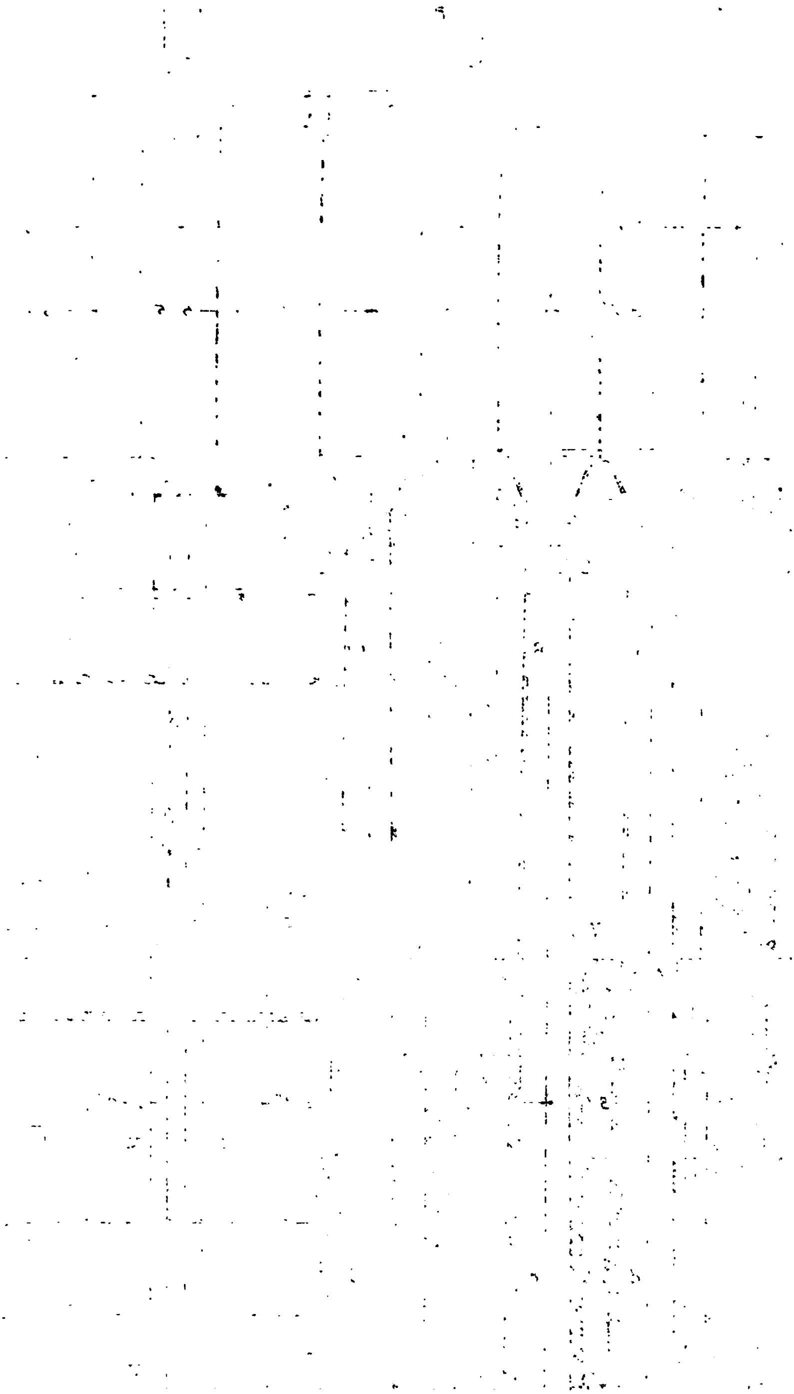


Plate VI B represents an end section of a bench of the improved Henderson or "Broxburn" retorts, showing two retorts in section. One malleable iron hopper (1) is provided for each retort. These hoppers have a capacity of about 54 cubic feet, and hold a supply of broken shale sufficient to run the retort for about 18 hours. The cast-iron portion of the retort (6), is of rectangular cross-section, 2 feet 9½ inches by 1 foot 2⅜ inches at the top, increasing with constant taper to 3 feet ¼ inch by 1 foot 5¼ inches at the bottom, where it is joined to fire-brick section (8) by fire-clay joint (7). The length of the iron part of the retort is 14 feet. The fire-brick part of the retort is made up of specially shaped brick, and the length of this part is usually 20 feet. Its cross-section is rectangular, measuring 3 feet ¼ inch by 1 foot 5¼ inches at the top, where it is joined to the iron part. At the bottom the cross-sectional measurements have increased to 4 feet 8 inches by 1 foot 10 inches. The shale in the retort rests on a pair of toothed rolls (9), about 4½ feet long, at the bottom of the retort. In operation, these rolls rotate slowly towards each other at a regulated rate, thereby permitting a controlled throughput of shale. Spent shale is discharged continuously by the rolls into hoppers (10), one for each retort. These hoppers are discharged periodically into cars operating on tracks (25) running beneath the retort benches. The overall height of the retort bench is about 65 feet.

Vapors pass out of the tops of the retorts through vapor-lines. 3, into 30-inch headers, 5, into condensers as subsequently described (Pl. IX). A slight vacuum is maintained in the retorts by some form of exhauster, often a fan, in the condenser system. Steam is admitted into the bottom of the brick part of the retort in about the same proportions as indicated in connection with the Young and Beilby retort, and serves the same purposes. Practically all of the oil is distilled from the shale in the cast-iron part (6) of the retort, at a temperature of about 900° F. The oil-spent shale reaches a maximum temperature probably in excess of 1500° F. in the bottom of the fire-brick part (8), but is discharged from the hoppers in a relatively cool condition, having been cooled by the incoming steam.

The new Henderson retort puts through 4 to 4½ tons of the shale now being mined in Scotland per day of 24 hours. The shale remains in the heat of the retort between 24 to 30 hours, and thus is heated up very gradually. The retort shows a slightly increased yield of oil over the older types, and a notable increase of ammonium sulphate. On account of the greater length of the retorts, depreciation is slower, and the life of a retort is from 7 to 10 years.

These retorts are heated by the fixed gases resulting from distillation of the shale supplemented by producer gas made from

dross coal, after both gases have been scrubbed for gasoline and ammonia. Four retorts are set in one oven, and 16 ovens comprise a bench, 2 retorts wide and 32 long. At the crude-oil works of the Broxburn Oil Co. Ltd., 3½ benches, or 224 retorts, of this type are in operation. The Henderson retort of 1889[78] differed from the improved type, mainly in that it was shorter, and the discharge mechanism consisted of one roll instead of two.

THE YOUNG AND FYFE RETORT.

Young's Paraffin Light & Mineral Oil Co. Ltd., uses a modified Young and Beilby retort called the Young and Fyfe.[79] This type is the old Young and Beilby retort made continuous, but inasmuch as its efficiency is lower and operating costs higher than both the Henderson and the Pumpherston, no detailed description is given here. The top hoppers are provided with rocking shafts to which chains are attached to facilitate the regular passage of raw shale into the retorts. Spent shale is discharged by a type of double screw into a large combustion chamber at the bottom of the retort. In this chamber the residual fixed carbon of the shale is burned out, but apparently this does not increase the efficiency of the retort or decrease the cost of fuel to any appreciable extent.

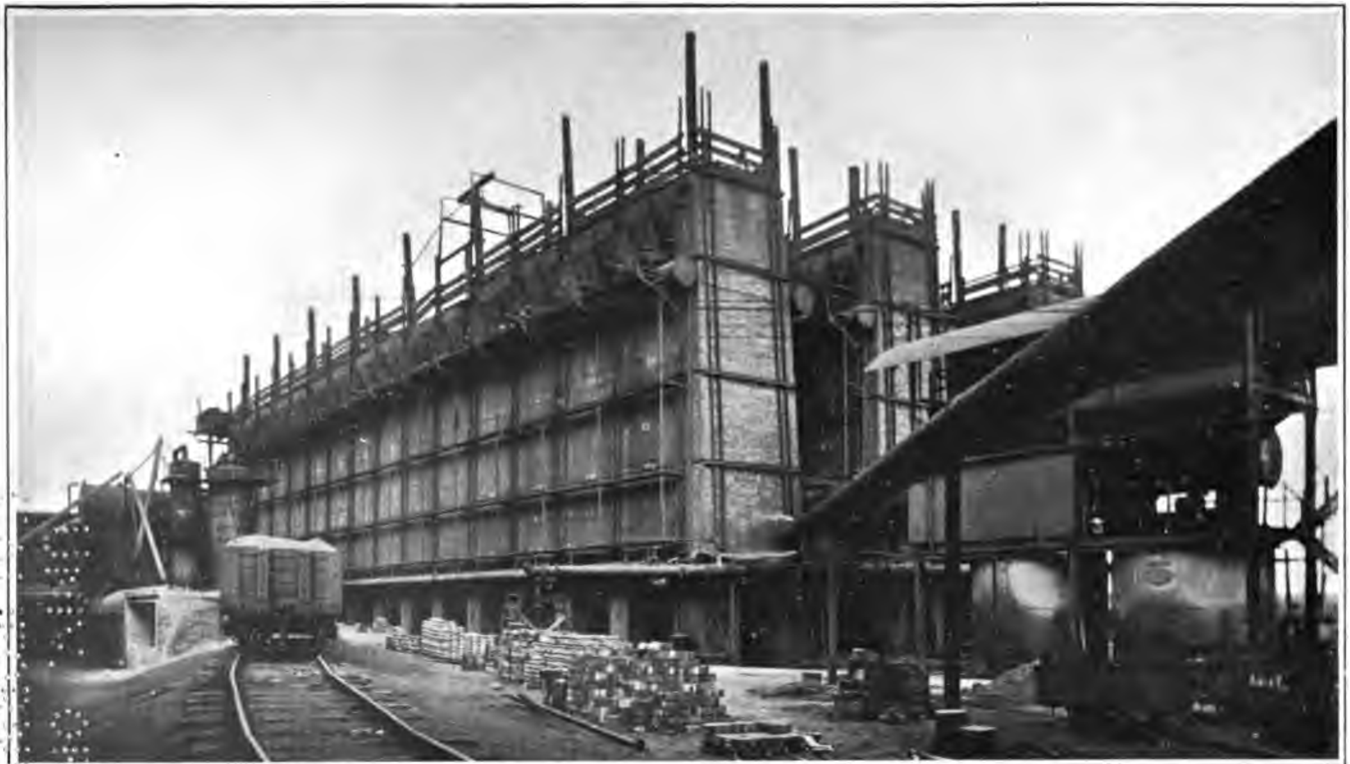
THE PUMPHERSTON RETORT.

The Pumpherston or Bryson retort, invented by Bryson, Fraser and Jones, of the Pumpherston Oil Co. Ltd.,[80] is undoubtedly the most efficient and best retort that has been devised to treat Scotch oil-shales. Plate VII shows the external appearance of this type of retort, which is used exclusively by the Pumpherston and Oakbank Oil Companies Ltd., both now part of the Scottish Oils Ltd. The Pumpherston company was, before the consolidation, the largest oil-shale company in Scotland, and during the past decade has paid the largest dividends of any of the present six producing oil-shale companies.

The Pumpherston retort is vertical and circular in cross-section. It consists of five essential parts (See Pl. VIII A); a hopper **a**; a cast-iron part **b**; a fire-brick retort part **d**, joined to **b** at **c**; a discharge mechanism **e**; and spent shale hopper **f**.

Hopper **a** (one for each retort) is of wrought iron and holds a supply of shale, such that the retorts need not be charged during the night or over Sunday. The top or cast-iron part of the retort is 11 feet high, circular in cross-section, tapered, 2 feet in diameter at the top and 2 feet 4 inches at the bottom. This part is a single casting, about one inch thick. At the top is an 8-inch vapor offtake **j**, cast integral with the retort and through which distillation products of the shale pass from the retorts into vapor-header **k**.

78 English Patent 6,726 (1889).
79 English Patents 13,665 (1897) and 15,236 (1899).
80 English Patents 8,371 (1894); 7,113 (1895); 4,249 (1897).



(Courtesy Scottish Oils, Ltd.)

BENCH OF PUMPH ERSTON RETORTS

The angle between the retort and vapor line **j**, is 45°, and a lip **l**, keeps the shale from clogging the off-take.

At **c**, the cast-iron part of the retort rests on, and is joined to, fire-brick part **d**. The joint is made with a special fire-clay mixture. The fire-brick part **d**, is 18 feet high, of circular cross-section, and like the iron part, tapers at a constant rate from top to bottom. It is 2 feet 4 inches in diameter at the top where it joins the iron part, 3 feet in diameter at the bottom, and is made of a single tier of brick.

The brick section is constructed of specially shaped fire-brick, made of exceptionally fine fire-clay found in the vicinity of the shale fields. The bricks are laid with a thin coat of fire-clay. Referring to the drawing (Plate VIII A), at points **z** will be noted special bricks extending from this fire-brick part, and touching similar bricks **m**, in the outer wall of the retort. These bricks are not tied to each other, but are free to move and expand and contract, but provide a support for the retort itself. The whole retort is in no way tied to the outer wall at any place, but each retort is free to expand and contract as a unit, without putting strain on any part of the bench. Just above the bottom of the retort is constriction **n**, below which is a circular iron table **o**, which supports the shale in the retort. Above this table is a curved arm **p**, which revolves above the table, being driven by a system of ratchets and pauls as indicated in Plate VIII B (x). In revolving, this arm thrusts the spent shale from the retort into spent-shale hopper **f**, common to two retorts.

Since the shale falls apart and thus expands about 25 per cent in volume during retorting, and also tends to stick and bridge, the retorts are tapered, as noted, to permit the shale to move freely.

The revolving arm ordinarily makes four revolutions an hour, but its rate, and consequently the rate of discharge of spent shale, can be regulated. The arm, besides discharging spent shale from the retort, gives the shale a certain amount of movement in the retort and discourages any tendency towards bridging, clogging, or sticking of the shale. Hopper **f** is discharged periodically into spent-shale cars by lowering bell **s**. It is to be noted that one track beneath the bench of retorts takes care of the spent shale from all of the retorts of the bench, due to the design of the discharge hoppers.

Steam—exhaust from the power plant—is admitted into spent-shale hoppers at **t**, and passes up into the retorts. Gas—used as fuel—enters combustion spaces at the bottom of the brick section, and the combustion chambers for this part are divided into four segments by baffle walls, which cause the flames to circle the outside part of the retort. Usually the hot gases from the brick section pass to the top of the oven **v**, surrounding the iron parts,

and strike an arch **w**, which directs them downwards into a flue **y**, starting at the bottom of the housing for the cast-iron section. Two smoke stacks **r**, serve a unit of four retorts, which latter are set in a common furnace; each unit of four is separated from the next unit by fairly thick brick walls. Sixteen units constitute a bench of retorts, two retorts wide and thirty-two long.

The Pumpherston retort proper holds 4½ tons of shale and its present through-put of shale is 4 to 4½ tons a day. Therefore a piece of shale requires 24 hours to pass from top to bottom of the retort. Inasmuch as the raw-shale hopper at the top of the retort holds 4½ tons in addition, a piece of shale requires 48 hours to travel from raw to spent-shale hopper.

In operation, freshly-broken shale is fed from time to time from cars **u**, (Plate VIII A) into hopper **a**, from which it gradually passes into the iron part **b**, in which practically all of the oil is distilled at a temperature probably not exceeding 900° F. The shale then passes into the brick part of the retort **d**, where it is subjected to the action of steam and reaches a final temperature certainly not in excess of 1800° F. (This temperature is reached in the lower part of the combustion chamber.) The amount of steam used varies with the quality of the shale, but usually amounts to about 100 gallons of water—as steam—to each ton of shale treated, that is, to each 25 gallons of oil produced.

The spent shale discharges into the spent-shale hopper **f**, and is finally dumped from the latter as described, into cars **g**. The spent shale usually contains only from 1½ to 2 per cent of fixed carbon, which could be removed by action of heat and steam, but this amount is allowed to remain as the economic limit as regards fuel consumption, through-put of shale, and ammonia and gas yield. Probably a greater yield of ammonia and gas could be obtained per unit weight of shale, by reducing the amount of fixed carbon in the spent shale, with more steam and longer retorting time, but at the expense of through-put and operating costs.

OPERATING TEMPERATURES.

Temperature measurements have never been made within the retort itself. A series of heat measurements in different parts of the combustion chambers immediately surrounding the retorts gave the following temperatures, which can probably be considered as averages:

Section of furnace	*Degrees F.*	
Bottom	1800	
Middle of brick part	1650	
Top of brick part	1500	
Middle of iron part	1100	
Top of iron part	900-1350	Average.....1100

The fires and temperatures in the different zones of the retorts are controlled by one man per shift, who determines retort tem-

peratures—visually—through peep-holes at various levels of the retort.

CAPACITY OF THE RETORT.

It has been stated that the capacity of a Pumpherston retort is 4 to 4½ tons of shale per 24-hour day. This is true, as regards the shale now being treated, but experiments have indicated that the capacity of the retort is one of oil production, rather than of shale through-put. It may be stated that the maximum daily capacity of a Pumpherston retort is about 96 gallons of oil, when operating under best conditions, that is, producing all the ammonia possible and the greatest yield of the best oil possible (this does not include scrubber naphtha). Therefore, in round figures, a Pumpherston retort will put through 10 tons of 10-gallon shale, and one ton of 100-gallon shale a day. Table 13 indicates this general tendency, the figures being the result of actual test runs.

TABLE 13—Capacity of Pumpherston retorts—effect of oil yield of shales on through-put. a

Yield of shale gallons per ton.	Through-put of shale, tons per day	Oil yield per retort per day, gallons.
16.1	3.42	72.7
19.5	4.25	81.4
24.2	3.81	92.0
29.6	4.32	129.5
28.2	4.32	121.2
35.4	2.63	92.7
61.4	3.36	207.5
135.8*b*	1.45	197.3

a Ammonia production was affected in a similar manner.
b Australian shale; through-put very slow, as it tended to intumesce and sinter in the retort.

LIFE OF THE RETORTS.

It has been stated that the life of a Pumpherston retort is over 20 years, as many have now been working continuously for that time. The average life of this retort is probably not so long, but with minor repairs it is well over 12 years. The cast-iron parts fail by overheating and swelling, which later produces cracking, and the brick part fails by cracking and wearing out. The scouring action of the shale in the lower part of the retort is sufficient slowly to wear this part thinner, hence the bricks used are selected more for their ability to resist abrasion than for their heat conductivity. Much care must be taken in starting and operating the retorts to avoid sudden heat changes, local overheating, or explosions.

LABOR FOR RETORTING.

A bench, or 16 units of four retorts each, 64 retorts in all, is operated by one charger, one dropper, one tip man, and one man

to clean pipes and to regulate heats, etc., a total of 4 men a shift, or 12 men a day. In May, 1920, the shale workers were granted a 7-hour day, in place of 8 hours.

REPAIRS.

As a unit consists of four retorts, when one is being repaired the four must be shut down. After repairing one retort, about 24 hours is required to heat the unit up to operating temperature. If a bench is shut down, the time required for heating up is two weeks. When a retort has been off for repair, it is heated slowly and preferably on spent shale, but frequently fresh shale is charged in at once, and distillation commences immediately. In charging an empty retort, care must be exercised to prevent disastrous explosions. Vapor mains are usually provided with explosion doors to provide for such accidents.

USE OF STEAM IN RETORTING.

As stated before, all modern Scotch oil-shale plants submit their shale to the action of steam in the retorts at a relatively high temperature. The steam serves many purposes, and its use in these retorts is essential. Exhaust steam, entering the bottom of the retorts, cools the hot spent shale passing from them, and in so doing the steam becomes superheated, and, passing into the retort, takes back to the latter much heat which would otherwise be wasted. The steam also distributes the heat evenly throughout the shale in the retort, thus helping to produce uniform temperatures. The steam sweeps the oil vapors out of the retort before they can decompose to any great extent. (See, however, page 182.) At the temperature of the base of the retort, 1300 to 1800° F., steam reacts with the fixed carbon of the shale to produce a mixture of carbon dioxide, carbon monoxide, and hydrogen. The two latter are combustible gases, valuable as fuels, and are made by the action of steam on the fixed carbon of the shale, which otherwise would not be utilized.

It is probable that some ammonia is produced from shales at low temperatures, possibly by the decomposition of amines and the like; also when the carbon is removed from the shale by the above reaction, much of the nitrogen of the spent shale is either set free in such condition that it can combine with the hydrogen produced by the interaction of steam and carbon; or more likely amino or imino compounds are freed, which are reduced to ammonia by the hydrogen. The nitrogen and hydrogen interacting, or the reduction of the nitrogen-containing compounds, produce ammonia, which, at the temperature of that part of the retort, would decompose completely into its elements, were it not that it is protected by excess steam and hydrogen, and the excess gas and steam immediately sweep it into a cooler zone. Under proper conditions, practically all of the nitrogen of oil-shales can

be converted into ammonia by the use of a large excess of steam and relatively high temperatures; but in practical retorting conditions in Scotland only about 60 per cent of the nitrogen in the shale is converted into ammonia.

The yield of paraffin in shale-oils has been increased experimentally by Irvine[81], who passed ammonia into the retorts. A possible explanation is that part of the ammonia decomposed, giving free hydrogen, and the increased concentration of hydrogen in the oil-producing zone inhibited, to a certain extent, the decomposition of the shale-oil. It seems hardly possible that hydrogenation of the oil takes place in the modern Scotch retort.

CONDENSATION AND SEPARATION OF PRODUCTS.

Large and comparatively low-speed rotary suction fans pull the vapors and gas from the top of the shale retorts, and into the condensing and scrubbing system. These fans are placed between the last (oil) scrubbers and a large two-foot gas header, to which the separate retort gas burners are connected; that is, the fans draw the vapors and gas from the retorts, through the condensers, water scrubbers, and oil scrubber, and deliver the gas into the burner supply header. Gas holders are not used, since the total volume of vapor-pipes, condensers, scrubbers, and gas header is large enough to take care of pressure fluctuations. The suction of the fans should not be more than 9 or 10 inches of water at the fan, but at the retort outlet it is ordinarily not over 5/16 inch. The whole retorting system, including retorts, vapor-lines, condensers, and scrubbers, thus operates under a slight negative pressure. The suction is naturally greatest at the fans, and least at the outlets of the spent-shale hoppers. Hence, in general, any leaks in this system are due to air leaking in, rather than vapors and gases leaking out. Large leaks are detected by gage readings, and by an increase of the nitrogen content (nitrogen from the air) of the gases coming from the scrubbers.

From the retort the vapors and fixed gases pass into large vapor-mains, usually 30 inches in diameter (the size varies however with the number of retorts connected with it), which conduct the vapors to the condensers. Sometimes boiler feed-water heaters, serving as economizers, are placed in the vapor-mains, but this practice is not general.

The condensers (Plate IX) are air-cooled and vertical. They consist of cast-iron pieces 9 feet long and 4 inches in diameter, making an inverted U at the top, and fitting into headers at the bottom. The condensers are four joints of pipe and about 40 feet high. For a 110-ton shale plant, 1392 cast-iron pipes are used, in

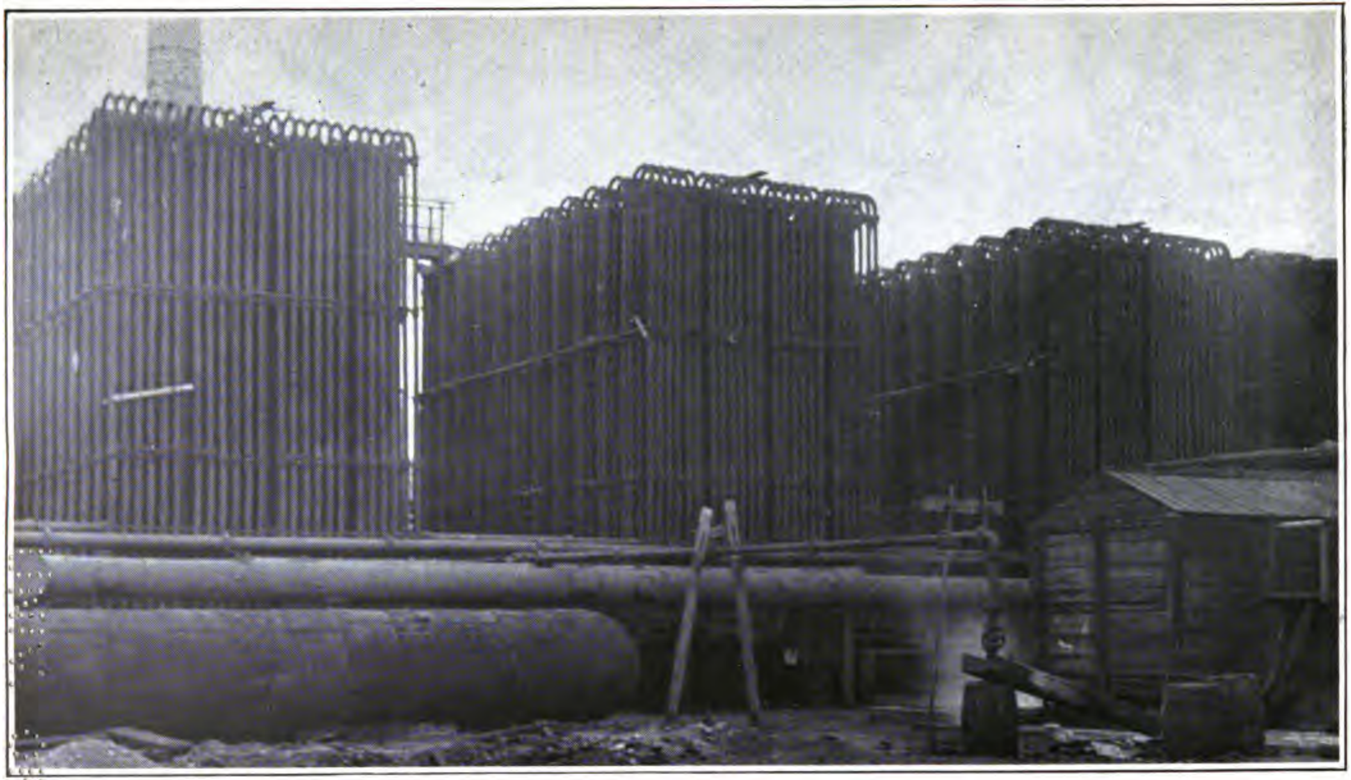
81 Steuart, D. R. The oil-shales of the Lothians, Part III, The chemistry of the oil-shales: Mem. Geol. Survey, Scotland, 2d Ed., 1912, p. 177.

addition to the headers, giving a total of 16,200 square feet of cooling surface, in addition to headers and return U's. In general, 15 square feet of cooling surface is used in Scotland for 1000 cubic feet of vapors and permanent gases per 24 hours. About 147 square feet of cooling surface is provided for each short ton of shale retorted per day. This is suitable for Scotch shales yielding up to 38 gallons of oil per ton. Generally a tubular water-heater is installed between the gas mains and condensers.

The condensers separate from the vapors and fixed gases the bulk of the oil and all of the water produced in distilling the shale, also shale dust. The water and oil flow from the condensers to separating boxes or tanks, where they are separated by gravity settling. The water, which contains in solution nearly all of the ammonia produced from the shale, together with certain fixed nitrogen compounds formed in retorting, flows into a tank from which it is piped to the ammonium-sulphate plant. The crude oil flows to another tank, from which it is either piped to the refinery or shipped there in tank-cars, depending on the distance of the refinery from the retorting plant. The crude has a high congealing point, 84 to 92° F., due to its high content of wax, and thus its transportation in pipe-lines is attended with difficulties. In some plants the oil flows in a jacketed line to the refinery, the inner pipe carrying the oil and the outer pipe the hot water recovered from the condensers on its way to the ammonia plant. The ammonia plant is commonly operated in connection with the refinery.

The gases passing from the condensers still contain small quantities of ammonia and light hydrocarbons not removed by the condensers. In order to separate these, the gases are passed into vertical scrubbing towers, in the first of which they are scrubbed with water to remove ammonia; and in the second, with an intermediate oil, which removes the lighter hydrocarbons (scrubber gasoline or naphtha).

The number and size of scrubbers varies with the size of the plant. As a rule they are vertical towers about 30 feet high and 6 feet in diameter. At a retorting plant there are usually two or more such towers for water scrubbing, and two or more for oil scrubbing. The towers are partly filled with coke or wooden checker-work, to insure intimate contact of the washing menstruum with the gases. The gases enter the towers at the bottom and scrubbing oil at the top, and gases leave the towers at the top and oil at the bottom. In the first towers, the gases are thus washed with water, and thereby ammonia, amounting to about 3 pounds of ammonium sulphate to a ton of shale retorted, is recovered. From the scrubbers, the ammonia-containing water is



(Courtesy Scottish Oils, Ltd.)

ATMOSPHERIC CONDENSERS FOR CRUDE OIL

piped to the sulphate plant, where it is combined with the condenser water.

In the oil-scrubbers the gas is washed with an intermediate oil, a fraction of shale-oil distilling between kerosene and the first lubricating fraction, and about 2.4 gallons of gasoline are thus recovered from the gases of a ton of shale retorted. From the scrubbers, the scrubber-oil, containing the dissolved gasoline, flows to continuous steam stills, in which the gasoline is distilled from the oil. The latter is then cooled and re-used for scrubbing, the same oil being used successively. The scrubbing of shale gases for gasoline is similar to methods used in the United States for obtaining gasoline from natural gas by the absorption method.[82]

Sometimes the fan is placed between the water and oil scrubbers, but usually the permanent gases pass from the scrubbers to the fans and are forced under low pressure into the large header, which leads them back to the retorts where they are burned as fuel in the furnaces.

PRIMARY OR CRUDE PRODUCTS.

At this stage the crude products obtained are permanent gases; crude oil, with which may be included scrubber naphtha; ammonia water, from scrubbers and condensers, and spent shale.

SHALE GAS.

Shales treated in Scotland in 1921 produced about 9,800 cubic feet of gas per ton retorted. A typical analysis of the gas is given in Table 14.

TABLE 14—Typical analysis of gas produced in retorting Scotch oil-shale.

Constituent	*Per cent*
Carbon dioxide (CO_2)	20.00
Carbon monoxide (CO)	4.28
Hydrogen (H_2)	34.21
Methane (CH_4)	10.80
Olefines (C_nH_{2n})	3.06
Oxygen (O_2)	5.10
Nitrogen (N_2)	22.55
Total	100.00*a*

Calorific value, gross, 302.5 B.t.u. per cu. ft.

Calorific value, net, 271.4 B.t.u. per cu. ft.

a The gas always contains a trace or more of hydrogen sulphide (H_2S).

82 Dykema, W. P., Recent developments in the absorption process for recovering gasoline from natural gas: Bull. 176, Bureau of Mines, 1919, 90 pp.

CRUDE OIL.

Crude oil obtained from the shale now being worked in Scotland (1921), amounts to about 24.5 gallons of oil (including 2.4 gallons of scrubber naphtha) per ton treated. The crude has a specific gravity varying from 0.870 to 0.895, (30.9 to 26.4° B.) and a setting or congealing point of about 84° to 91° F.

The oil is green, greenish-brown, or brown in color, and is a complex mixture of organic compounds. (For the known chemical constituents of shale-oil see page 90.) It has a peculiar odor, faintly resembling garlic, but not particularly disagreeable.

AMMONIA WATER.

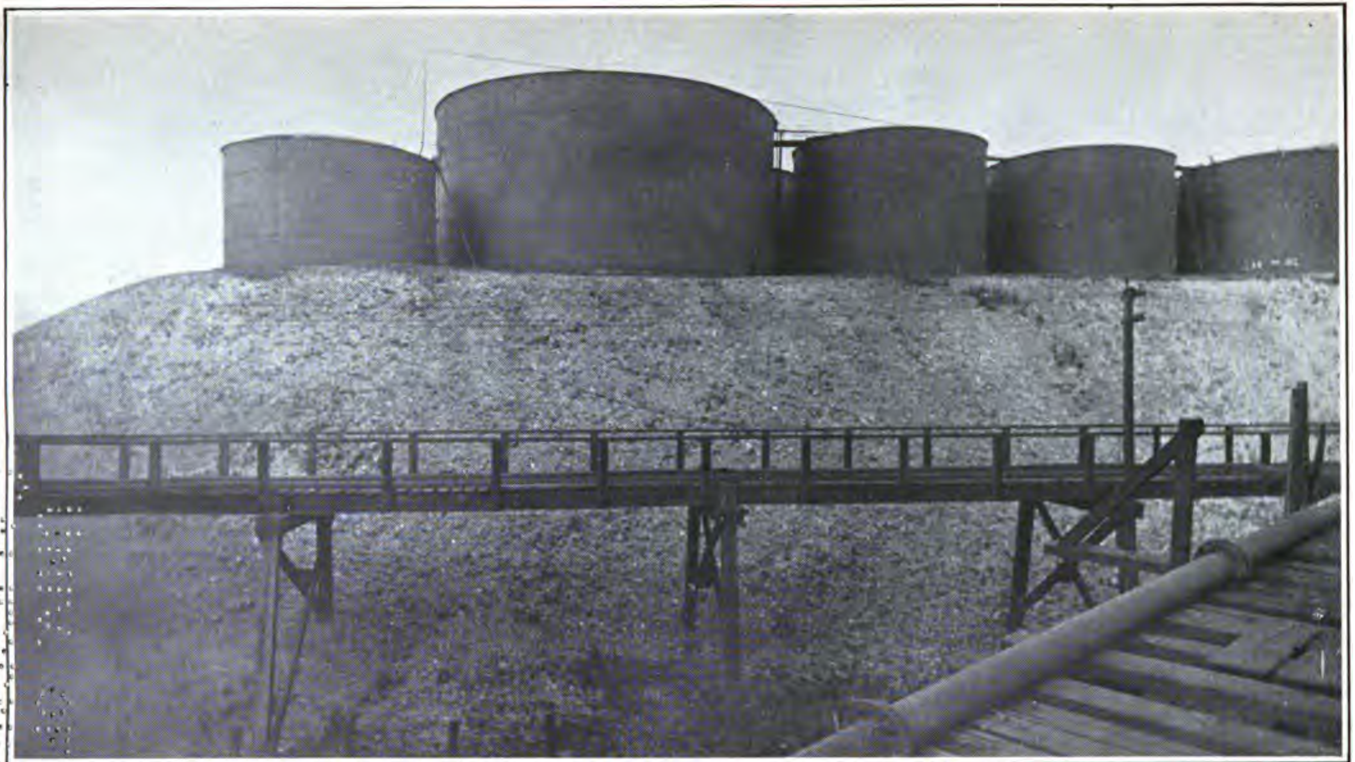
The ammonia water from the condensers and scrubbers is yellowish in color, and usually has an odor of pyridine or quinoline. In addition to dissolved ammonia it contains fixed nitrogen-containing compounds, such as pyridines, cyanides, and the like. In 1919, the shale treated in Scotland produced about 35.7 pounds of ammonium sulphate per short ton. This represents about 60 per cent of the total nitrogen in the raw shale.

SPENT SHALE.

The spent shale, or residue from the retorts, amounting to 75 or 80 per cent of the weight, and 125 per cent of the volume of the raw shale treated, has no value. Its removal involves an expense, and in many cases land on which to dump it must be leased. All the Scotch plants are marked by immense spent-shale dumps or "bings". (See Plate X.)

Small particles of spent shale are black, because of their fixed carbon; large pieces are bluish-white on the edges, where the carbon has been completely removed. The fixed carbon in the spent-shale dump slowly oxidizes, and the small amount of iron present changes from the ferrous to the ferric form; thus the color of the bing slowly changes from grayish-black to a reddish color, and because of these reactions the dumps remain warm and give off an unpleasant odor.

Spent shale has been tried for road and brick-making in Scotland, with poor results. The colloidal properties of the silica in the shale have been destroyed by the heat of the retort, consequently spent shale has no value for making brick even when it is oxidized, as the binding properties of the clay are entirely destroyed.



(*Courtesy Scottish Oils, Ltd.*)

STORAGE TANKS ON SPENT SHALE "BINGS"

(Courtesy Scottish Oils, Ltd.)

SCOTCH SHALE-OIL DISTILLING PLANT

YIELD OF PRODUCTS.

Table 15 shows the crude or primary products yielded by one ton of Scotch oil-shale of the kind treated in 1919 (retorted with 100 gallons of water, as steam.)

TABLE 15—Crude products from one ton of Scotch oil-shale (1919).

Product	Quantity	Properties	Disposal
Gas	9800 cubic feet	Heat value 270 B.t.u. per cubic foot	Used as fuel in retort furnaces.
Crude oil *a*	24.5 gallons	Specific gravity 0.860 ; setting point 86° F.	Sent to refinery.
Ammonia water *b*	35.7 pounds		Sent to sulphate plant for making ammonium-sulphate
Spent shale	1500-1600 pounds		Dumped as waste.

a The crude oil includes scrubber naphtha. The crude and scrubber naphtha are not ordinarily mixed, and are kept separate during refining operations. If mixed in proper proportions, however, the specific gravity of the mixture is as given. The specific gravity of the crude oil alone is about 0.885.

b Expressed in terms of equivalent $(NH_4)_2 SO_4$.

REFINING SHALE-OIL.

The refining of shale-oil in Scotland is similar, in general principles, to the refining of petroleum in other parts of the world, but is more involved than the equivalent refining of petroleum, because shale-oil is, chemically speaking, more reactive than the average petroleum, and it contains a greater percentage of those substances that must be removed in the refining process to produce marketable products. Shale-oil refining is a more costly process than petroleum refining, chiefly because of this difference in the chemical nature of the two oils.

The most valuable product derived from Scotch shale-oil has always been paraffin wax. Therefore, conditions for retorting the oil-shales are such as will produce, within economic limits, an oil containing the maximum amount of wax, and refining operations are such as will produce, as cheaply as possible, the maximum amount of marketable wax from the crude oil.

A Scotch shale-oil refinery differs from the ordinary American petroleum refinery of equal capacity, in having (1) a much greater number of small stills, a large proportion of which are intermittent or batch stills; (2) a greater number of small and often horizontal agitators; (3) an extraordinarily large wax plant; (4) a separate condensing tank for each still, and (5) most of the oil in various stages of refining is handled by compressed air instead of pumps. This necessitates the use of strong horizontal cylindrical tanks, many of them on the ground level. Another difference noticeable to the petroleum refiner is the absence of "look-boxes" for watching the flow of distillates in the delivery

lines from condensers. The streams of distillates are always in sight, however, as they flow from the condensers to the distributing lines. Plate XI gives a general view of the distillation plant of a Scotch shale-oil refinery.

Very little is known regarding the actual chemical composition of shale-oils (page 90). However, they contain a large percentage of compounds which must be removed before the products are of commercial grade. Probably most of these compounds are members of the various unsaturated series of hydrocarbons, phenols and the like; and various nitrogen-containing bodies, such as homologues of pyridine. Regarding the latter, although the effort is made to fix as much as possible of the nitrogen of the shales as ammonia, yet undoubtedly the crude oil contains a considerable percentage of nitrogen compounds. This is evidenced by the fact that a 10 per cent sulphuric acid solution will remove about 10 per cent of the volume of the crude oil. The objectionable substances mentioned are removed by treatment with sulphuric acid and caustic soda or soda ash, so most of the refinery loss is in the form of acid and alkali tars and sludges. Because of the high percentage of objectionable compounds, acid and alkali treatments are more numerous than in ordinary petroleum refining, and re-distillations of unfinished products are more frequent.

Plate XII shows graphically the ordinary procedure at a shale-oil refinery.[83]

The chart shows that the crude products of shale distillation are ammonia liquor, crude oil, gas, and spent shale. The gas, as before mentioned, is scrubbed for ammonia and naphtha, and the scrubber oil, containing the naphtha, is distilled and crude naphtha recovered therefrom. The crude naphtha is then treated with acid and alkali and re-distilled, giving various grades of finished naphtha and still bottoms, which are added to the crude burning oil from the crude.

Following the diagram it will be noted that the crude oil is first distilled, giving (1) coke, (2) a crude distillate (green oil), and (3) crude naphtha. The naphtha is treated with acid and soda, and yields (4) several finished naphthas, and (5) bottoms, which are added to the crude burning oil fraction of the green oil.

83 Some of the terms used in refinery procedure to describe the oils, such as "blue oil" and "green oil", date from the time of the first shale-oil refinery, that of Young, when much secrecy was thrown around the industry. These and similar terms were used to mislead the curious.

The green oil (2) is acid and soda-treated and again distilled, yielding (1) coke, (6) crude burning oil, to which is added (5) bottoms from crude naphtha distillation; and (7) heavy oil containing paraffin wax. The crude burning oil (6) is chemically treated and fractionated into (8) motor boat oil (a distillate fuel for internal-combustion motors of the Diesel type); burning oil (9); and light gas or distillate fuel oil (10). Burning oil (9) is again treated and yields the finished lamp oil or kerosene (11). The heavy oil carrying paraffin wax (7) is cooled in refrigerators, pressed in filter and hydraulic presses and yields blue oil (12) and hard paraffin scale (13), which is mixed with soft scale (22), obtained in further processing of the blue oil. The hard scale (13) is sweated in sweating houses (Plate XV) and yields an oil (14) which is added to the green oil (2), or crude distillate; wax mixed with strainings (15), which is added to more hard scale (13), and again sweated; and various grades of waxes (16), which are melted and filtered through, or treated with, fuller's earth, after which they are ready for the market.

The blue oil (12) is treated with acid and alkali, and subsequently distilled to coke, yielding finished heavy burning oil (19); light gas oil (18); heavy gas oil (17); unfinished lubricating oils (20); and coke (21). A residual oil is also obtained and used in making greases. The heavy gas oil (17) is separately cooled and pressed, yielded finished gas or fuel oil (24) and soft scale (22), or wax, which is sweated, the final product being miners' wax (23); or the soft scale (22) is mixed with hard scale for the production of finished wax. The unfinished lubricating oils (20) are also separately treated in a similar manner, yielding like products, and lubricating oils (25), which are again treated with acid and alkali before being ready for the market.

The diagram and description are necessarily brief, and not entirely complete, as in many cases fractions overlap, as for example, fractionation of the crude burning oil yields a certain amount of heavy naphtha or gasoline, which is added to the naphtha previously produced. If the diagram and description indicate the complexity of shale-oil refining, they will have served their purpose.

The scrubber naphtha is handled separately, as may be noted on the diagram. On fractionation, however, it yields still bottoms, which are added to the crude burning oil.

Figure 3 shows diagrammatically the process of refining shale-oil, indicating the approximate percentage of products yielded from a ton of shale. The rectangles are intended to be in pro-

portion to the yields of products.[84] Plate XIII is a view of the largest oil-shale retorting plant in Scotland, and Plate XIV shows the typical lay-out of a complete retorting and refining works.

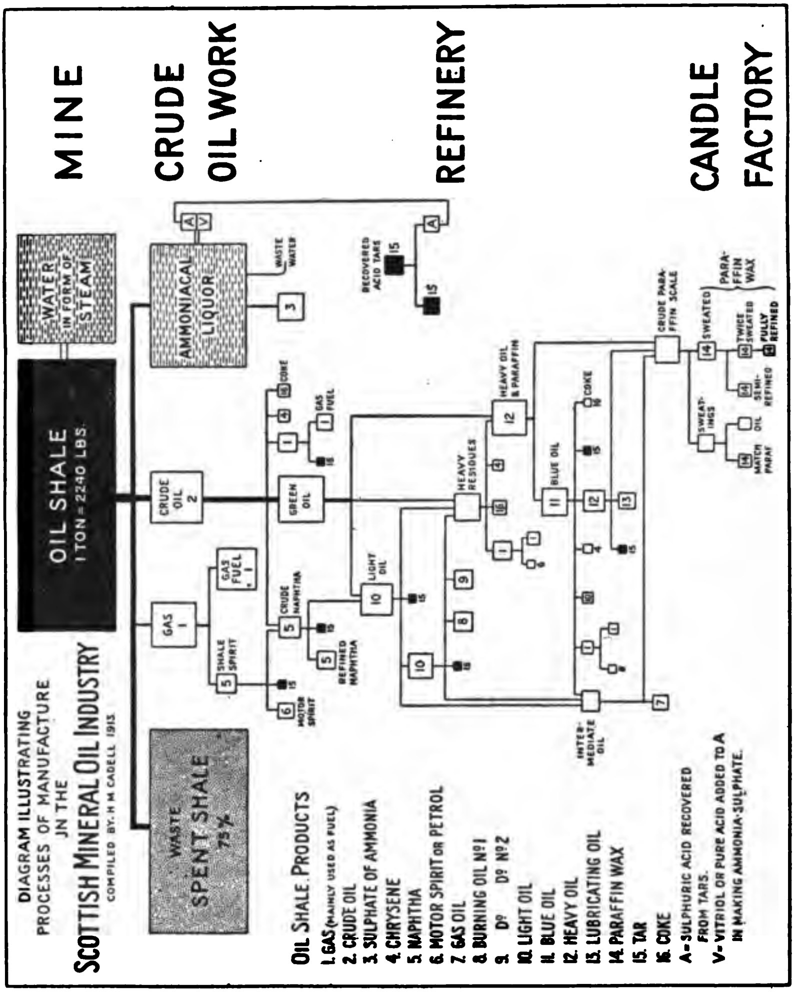


Figure 3

NOTES ON REFINING.

1. The crude oil is settled in tanks for 12 to 18 hours to separate water and shale dust before being run to the stills.

2. In the more modern refineries a combination of continuous and batch stills is used for fractionations. Some arrangement such as described below is often used. The center still of a

84 The writer is greatly indebted to Mr. H. M. Cadell for permission to use this diagram.

(*Courtesy Scottish Oils, Ltd.*)

GENERAL VIEW OF PUMPHERSTON WORKS, SCOTLAND
(Note spent shale "bings" in background)

battery of five stills receives the oil, and removes the lightest fraction. From this still the residue flows into similar stills on each side, which remove heavier fractions. These stills in turn feed to two more, where another fraction is removed. These finally deliver the residuum into heavy cast-iron pot or coking-stills, operating intermittently. Each battery of five continuous stills has six or more of the pot-stills, of which three or more are on each side of the continuous battery. The final continuous still discharges into one pot-still until the latter is full, and the stream is then switched to a second pot-still, and the first distilled down to coke. Meanwhile a third pot-still is cooling and being cleaned of coke, making ready to receive residuum from the continuous battery when the second pot-still has filled up. In the pot-stills the residuum is run to coke. The coke, amounting to about 2 per cent of the crude, is sold as fuel, or is used for the manufacture of arc-light carbons, electrodes, and the like (see, however, page 87). In many refineries the continuous battery consists of three stills instead of five. Sometimes only batch or pot-stills are used in refining the oil.

3. Steam is admitted to the bottom of the stills during all distillations. The quantity used, as condensed water, amounts to from 10 to 25 per cent of the total distillate, and averages 20 per cent. The steam is partly exhaust steam from the power plant. In some cases, rather inefficient heat exchangers, set in the still vapor-lines, are used to heat ingoing oil. Steam is passed into the still for three hours after the fire has been drawn. The only exception to the use of steam is in the pot-stills for a period just before coking takes place. A "cracking" of the oil is thus brought about, which yields a larger quantity of paraffin wax. The amount of steam used varies with the nature of the distillate being produced, and is usually greatest when the heavier fractions are coming over.

4. Gases evolved in the distillations are scrubbed for light oils, and are either used as fuel for heating retorts or stills, or, on account of their high illuminating and calorific value, for lighting. Gas from the stills amounts to over one cubic foot per gallon of crude treated.

5. The bottoms of the pot-stills are of cast iron, and are hemispherical in shape. The casting is about two inches thick. The cast-iron part is usually 8½ feet in diameter at the top and from 3½ to 4½ feet deep. The upper part, of steel, is of the same diameter and 6 feet high. Each still of this size has a water-cooled condenser of 225 feet of 4-inch cast-iron pipe. A still of this size charges about 2400 gallons of oil. The time taken in charging, distilling, cooling, and cleaning amounts to from 24 to 30 hours. The bottoms of the stills, with minor repairs, last about a year. A new bottom can be put on without much difficulty.

The continuous stills are of the horizontal boiler type, usually 8½ feet in diameter and 30 feet long. Their condensers are water-cooled, and provide about 850 feet of condensing surface.

6. The quantity of sulphuric acid used in refining amounts to about 3 per cent by volume of the crude oil. The strength of the acid is about 66° Bé (93 per cent H_2SO_4). Some of the refineries have their own acid-making plants. Agitators for partly refined products are small; few, if any, charge over 6000 to 7000 gallons of oil. The crude distillate (green oil), is treated in larger agitators, which charge as much as 50,000 gallons. Agitation is usually by compressed air. Acid of proper strength and proportion is usually added in batches, or sometimes all at once, and agitated from 15 minutes to an hour. Tar is allowed to settle for 2 to 18 hours, the average being about 6 hours, depending upon the grade of oil under treatment. The acid-treated oil is then run to soda agitators and similarly treated with a solution of caustic soda or soda-ash. The oils are not water-washed before distillation. Caustic soda is more commonly used than soda-ash.

It is impossible to go into detail as to the quantity and strength of chemicals used in treating the different oils, as these factors differ for nearly every fraction. An acid-tar from one treatment is often used as the first-acid batch in the treatment of another oil. Also, dilute acid recovered from the acid-tars is frequently used in treating. The strength of sulphuric acid used for various oils and in various stages of refining varies from 40 to 66° Bé (48 to 93 per cent H_2SO_4).

About 1½ per cent by weight of crude caustic soda, or its equivalent in soda-ash, is used in treating and refining; thus about 11 pounds of caustic is used per 100 gallons of crude oil. Certain distillations, especially of heavy oils, are made over caustic soda, and the strength and quantity of alkali solution used in treating varies with the nature of the fractions under treatment, but its specific gravity ranges from 1.01 to 1.38. Soda-tars from one treatment are frequently used in treating another oil.

7. Acid-tars are recovered, washed, and steamed to recover free acid. In this way, from 50 to 60 per cent of the acid used in making ammonium sulphate is obtained. The acid-free tars, as well as the soda-tars, are used as fuel under the stills. The tars or sludges obtained in refining amount to about 16 per cent of the crude treated.

8. Just before distillation ceases in the coking stills, a small amount of chyrsene, a peculiar reddish-colored product like resin, is obtained. It is not further purified except to float off water and oil adhering to it. It is usually considered a loss, but is sometimes used in laying wood-paving blocks.

9. Particular attention is paid to the recovery and treatment of the paraffin wax. The methods now generally used are largely

the inventions of James Bryson of the Pumpherston company, and the late N. M. Henderson of the Broxburn Oil Co. Ltd.[85] The method is essentially as follows, varying somewhat in different plants:[86]

The heavy oil is cooled in tanks in open sheds and subsequently by freezing machines, usually of the ammonia absorption type, or compressors with anhydrous ammonia, operating with calcium chloride brine. Some coolers are directly cooled with anhydrous ammonia, notably those at the Pumpherston and Oakbank works, where compressors are used. Cooling takes place slowly to preserve the crystalline structure of the wax as well as possible. The chilled mass of paraffin, now a pasty mixture of wax and oil, is then pumped into filter-presses, which are in refrigerated rooms. As a rule, low-pressure filters are used, so the wax cakes from these filters are then pressed in cloths in hydraulic presses, after which the wax is termed "hard scale". The oil separated from the wax is the "blue oil" referred to on page 81. The scale is now melted and run onto strainers of rather fine screen in long shallow trays. The wax is floated in the trays by water, but when it cools and solidifies the water is run off, leaving the cake of wax on the screen. The trays are built in special houses (sweating stoves), heated with steam coils, and then these houses are closed and sweating begun. The temperature is increased to such an extent that the oil and soft paraffin melt and run away from the harder wax, until the latter has the desired purity and melting-point. The wax remaining on the trays is then melted by direct steam and run to filters, filled with bone-black or fuller's earth. The sweatings are re-run for softer or lower melting-point wax.

Henderson also designed a sweating apparatus which is said to be an improvement on the tray types. This apparatus consists of several vertical cylinders 9 feet high and 17 inches in diameter, with a central cylinder 7 inches in diameter, open at top and bottom inside the outer cylinder. The warm wax is run into the space between the two cylinders and floated on water until it has solidified. The water is then run off and the cylinder slowly heated as in the other types of sweaters.

In the early history of the Scotch oil-shale industry, many processes were proposed and used for the recovery and treatment of wax. Improvements were all intended to insure better and cleaner recoveries, and to lessen plant and labor costs. The Bryson type of coolers, and Henderson's sweating stoves have practically superseded all other types. They are used in the United States and elsewhere under different names. Plate XV

85 English Patents 9557 (1884); 1291 (1887); and 11,799 (1891).

86 This method is followed in general by petroleum refiners the world over, who owe much to the Scotch oil-shale industry in this and other features of petroleum refining.

illustrates a typical installation of sweating sheds in a shale plant.

Much of the wax produced by the shale operators is made directly by them into candles, but the larger part is sold in bags or slabs to independent candle-makers.

PLANT ECONOMIES.

It is not out of place here to mention again the fact that certain practices in the Scotch oil-shale industry could well be adopted more generally by the American petroleum refiner. For example: acid-tars are treated, the free acid recovered and used in further treatment of products, or for the production of ammonium sulphate. Still gases are not wasted, as a rule, but are collected and used for illuminating or fuel purposes. Although much of the Scotch plant equipment may seem cumbersome, it well accomplishes its purpose, and plant operations are conducted with an eye to economy and prevention of losses and waste.

YIELD OF PRODUCTS.

Early in the history of the industry certain products were made and a market was created for them, and since then the operators have made no great effort to produce others. In fact, really little is known as to the possibilities of making products other than those now being made. With a definite market for their products, the operators strive to produce a crude oil that will yield the maximum of these products at lowest cost. Conditions have been fixed for producing such a crude oil, and it is known that if retorting conditions different from these were applied, a less satisfactory crude would be made, resulting in a smaller yield of refined products.

In 1919 the average yield of refined products from typical Scotch crude shale-oil was about that shown in Table 16, following:

TABLE 16—Yield of refined products from Scotch shale-oil in 1919

Products	Yield, per cent
Naphtha (gasoline), including scrubber naphtha, maximum end point 450° F.	9.9
Illuminating and burning oils	24.8
Gas and fuel oils	24.4
Lubricating oils	6.6
Paraffin wax *a*	9.5
Still coke	2.0
Loss *b*	22.8
Total	100.00

a The quantity of wax of various melting-points recovered amounts to about 17.7 pounds, or 2.4 gallons per ton of shale, or 9.5 gallons of wax obtained from 100 gallons of crude oil.

b Loss includes chrysene, tars, and permanent gases.

(*Courtesy Scottish Oils, Ltd.*)

SWEATING STOVES OR HOUSES FOR THE PRODUCTION OF WAX

The products, marketed under various trade names, are:

1. The **gasoline**, and **naphthas** (sold in three or four grades) are usually motor spirits, cleaners' naphtha, and solvent naphtha.

2. **Burning oils**:—A good grade of illuminating oil for domestic lamps is made, together with oil specially prepared for burning in lighthouses, railway signal lamps, and marine and agricultural oil-engines.

3. **Intermediate oils**:—(Gas and fuel oils): In this class are oils used for fuel purposes and for making and enriching gas. Much of this oil is sold as fuel for Diesel and semi-Diesel engines.

4. **Lubricating oils**:—Usually lubricating oils of three different specific gravities are made, all of which have low viscosities. They are usually sold unblended, but are sometimes mixed with animal or vegetable oils.

5. **Paraffin wax**:—This is used in making various kinds of candles and tapers. It also finds a use in the manufacture of matches, explosives, waterproof coating, impregnated paper, etc.

In addition to the above products, **still coke** is also made and sold as a fuel. The coke made from soda-treated oils is contaminated with soda salts (some distillations are made over soda solutions) and is usually not pure enough to be used for purposes other than for a high-grade fuel. Coke made in the primary crude distillation and certain other distillations is pure enough to be used for making electric-furnace electrodes and the like.

Soda and acid-tars: Inasmuch as these are used as fuels in the refineries, they can also be considered products, although they are not marketed. Formerly they were sold for grease making. A **residual oil**, often recovered in re-running the lubricating fractions, is used for grease making.

CHARACTER OF PRODUCTS

In Table 17 is given the properties of some of the refined products from Scotch shale-oil. Some of this information was obtained from operators, the remainder from tests made by the author on samples obtained from Scotland. For purposes of comparison, the distillation table of two typical American gasolines, meeting the 1920 specifications of the United States Government Committee on Standardization of Petroleum Products[87] is included in the table. Particular attention is called to the homogeneity of the Scotch products, as shown by distillations.

Table 18 is quoted from a Scotch authority as representing the properties of average samples of the usual products from Scotch shale-oil.

---

87 Bull. No. 3, Report of Committee on Standardization of Petroleum Specifications, 1919, 8 pp.

TABLE 17—Properties of Scotch shale-oil products.

	Motor spirit	Naphtha	Burning oil	Fuel oil	Lubricating oils		American Gasoline *e*	American Gasoline *e*
Specific Gravity	0.720	0.742	0.799	0.863	0.899	0.893	0.741	0.755
Baume Gravity *c*	64.4	58.7	45.2	32.2	25.7	26.8	58.9	55.4
Flash point °F.	....	...	103	240	360	334	...	...
Distillation *a* first drop, °C.	52	71	150	226	270	228	60	46
Per cent								
10	75	95	173	266	336	299	87	74
20	81	103	187	284	356	322	100	94
30	86	109	196	294	360	337	110	108
40	92	116	204	308	362	346	120	120
50	97	122	213	312	364	352	129	134
60	102	129	221	324	368	360	139	147
70	108	137	231	336	370	370	152	164
80	117	148	242	344	372	372	168	184
90	134	162	261	378	377	376	188	210
95	...	...	278	...	...	...	204	225
Dry *b*	147	180	285	382	384	378	224	225
Viscosity	...	...	...	...	215*d*	140*d*	...	...
Setting point, °F	...	...	...	...	26	25	...	...

a Engler-Ubbelholde Method on 100 cc.
b Losses not given.
c Modulus 140.
d Redwood at 70° F.—215 Redwood corresponds to about 258 Saybolt Universal and 140 Redwood to 165 Saybolt Universal. See also Table 19 (page 90) for comparative data on viscosities.
e Bureau of Mines method—see Tech. Paper 214, 1920, 33 pp. Sample meets 1920 Government Specifications.

TABLE 18—Properties of average Scotch shale-oil products.a

Product	Specific gravity	Flash point *b* range, °F.	Tint No. 500 glass Lovibond 2-in. cell	Boiling point *c* range, °F.	Viscosity, *d* secs.
Motor Spirit	0.715		Colorless	120-300	..
Spirit or Naphtha	0.735	Lowest Temp.	Colorless	140-320	..
Spirit or Naphtha	0.740		Colorless	180-380	..
Special oil or water white oil	0.785	106-120	..	310-500	..
Crystal oil	0.800	115-125	0.5	340-575	..
No. 1 burning oil	0.805	110-135	0.75	310-580	..
Lighthouse oil	0.810	145-160	1.0	390-573	..
Mineral or marine sperm	0.830	200-230	2.0	450-675	..
Mineral Colza	0.840	220-240	5.0	500-700	..
Gas oil	0.855	200-230	..	450-710	..
Gas oil, grease oil, cleaning oil	0.870	275-300	..	500-720	..
Lubricating oil	0.865	275-300	15	575-710	44
Lubricating oil	0.875	300-310	18	575-800	47
			½-inch cell		
Lubricating oil	0.885	320-340	15	650-870	55
Lubricating oil	0.895	330-350	16	720-875	70

a From Steuart, D. R. The oil-shales of the Lothians, Part III, The chemistry of the oil-shales: Mem. Geol. Survey Scotland; 2d ed., 1912, p. 191.
b Abel apparatus—closed cup for light burning oils; open cup for others.
c Probably Engler-Ubbleholde method.
d Viscosimeter not specified. Companies in 1912 each had own standard viscosity pipette.

Naphtha—According to Steuart[88], sixty to seventy per cent of the naphthas can be removed by treatment with fuming nitric or sulphuric acid. However, in refining, the strength and quantity of acid used is so adjusted that, while all highly unsaturated compounds are removed, the bulk of the olefins remain in the finished products. This also applies to products other than naphtha. Thus, the writer found by testing a Scotch shale motor spirit for unsaturation, by the method used by the Bureau of Mines[89], that 22 per cent of the oil was removed by sulphuric acid (sp. gr. 1.84). Shale naphtha has been found to contain benzene and toluene[90]. B. Steuart[91] found the naphthenes, methyl-tetramethylene, pentamethylene, and hexamethylene, in shale naphtha. Heusler[92], in addition, found metaxylene and cumene in the same product.

The specific gravities of the shale naphthas range from 0.700 to 0.750 (70.8 to 56.7°B) or more.

Burning Oils. These are colorless, especially those prepared for domestic kerosene lamps. The heavier oils may, however, become more or less colored on exposure to sunlight. Shale naphthas and burning oils are characterized by a strong bluish fluorescence. From 30 to 40 per cent of the volume of the oil can be removed by fuming sulphuric acid[93]. The specific gravities

88 Steuart, D. R., work cited, p. 190.
89 Dean, E. W., and Hill, H. H., The determination of unsaturation in gasoline: Tech. Paper 181, Bureau of Mines, 1919, 25 pp.
90 Steuart, D. R., work cited, p. 190.
91 Steuart, B., Contribution to the composition of shale naphtha; Jour. Soc. Chem. Ind., vol. 19, 1900, pp. 986-989.
92 Heusler, F. "Uber der zusammazetzung der schottische schiefertherole. Ber. Deut. Chem. Gesell, bd. 30, 1897, pp. 2743-2752.
93 Steuart, D. R. The oil-shales of the Lothians, Part III. The chemistry of the oil-shales: Mem. Geol. Survey, Scotland 2nd ed., 1912, pp. 191-192.

of the burning oils vary from 0.790 to 0.830 (47.2 to 38.7°B), and flash points from 105 to 230° F.

Intermediate oils—Gas oils, etc., are used extensively in gas making and for fuel in internal-combustion engines. The British navy also uses large quantities for fuel. These oils are also sold for cleaning purposes. Their specific gravity varies from 0.840 to 0.870 (36.7 to 30.9°B).

Lubricating oils can hardly be considered viscous enough for high-duty work, but do possess the property of holding their viscosity well with rising temperature. The lubricating oils are sometimes blended with animal or vegetable oils, although most are sold unblended. The specific gravity of the lubricating oils ranges from 0.865 to 0.910[94] (31.8-23.9°B).

Table 19 below will be found of interest in this regard, as it gives the viscosities at different temperatures of various American lubricating oils particularly adapted for use in internal-combustion motors, as compared with the viscosities of a Scotch shale-oil lubricating oil at corresponding temperatures. It is unfortunate that the writer had but one sample of Scotch oil for the test, nevertheless the figures bear out the statements made in the preceding paragraph:

TABLE 19—Viscosities of petroleum and Scotch shale lubricating oils at various temperatures.

Oil	Saybolt viscosity at 40° C	60° C	100° C
Eastern extra heavy motor	1325	451	108
Eastern medium motor	502	195	63
Eastern heavy motor	326	136	53
Eastern light motor	164	81	42
Eastern light motor	132	71	41
Scotch shale	140	68	43

Paraffin wax is made in various grades, the usual melting points being 130-32°; 125-127°; 118-120°; 110-112°; and 98-102°F.

CHEMICAL COMPOUNDS FOUND IN SHALE-OIL.

Comparatively little is known as to the chemical constituents of shale-oil; however, its composition is known to vary with the nature of the shale from which it was obtained, and with the method by which it was produced. Inasmuch as shale-oil is produced by destructive distillation of substances that undoubtedly differ in different shales, this is self-evident.

Some studies have been made of chemical nature of shale-oil. In addition to members of the aromatic and naphthene hydrocarbons (page 89), Gray[95] found traces of phenol, ortho and meta-cresols, xylenols, and guaiacol in Scotch shale-oil. The

94 Steuart, D. R., work cited, p. 193.
95 Gray, Thos., The phenols from shale-oil: Jour. Soc. Chem. Ind., vol. 21, 1902, p. 845.

quantities present were too small to warrant their commercial extraction. Several investigators[96] have studied the basic nitrogen constituents of Scotch shale oil, and pyrrol (C_4H_5N); pyridine (C_5H_5N), and several of its homologues; and members of the quinoline (C_9H_7N) and isoquinoline (C_9H_7N) series have been isolated and identified.

AMMONIUM SULPHATE.

The ammonia water recovered from the retort condensers and scrubbers is pumped through a heater, in which it is heated by the spent or waste water flowing from the still, and passes into the top of a tower still, circular in cross-section, and approximately 30 feet high. Details of the ammonia stills vary in different plants but in general they have a series of cast-iron trays about 2 feet apart from top to bottom[97]. Steam, at a pressure of from 30 to 40 pounds, is admitted to the bottom of the tower and passes upwards through sealed hoods, cast on the trays, the liquid, flowing downward, passing from tray to tray by overflow traps. The steam carries off the volatile ammonia and, in addition, milk of lime is added about half way down the tower, breaking up and setting free the ammonia contained in fixed compounds from which it cannot be removed by simple steaming. Sulphur compounds in the water also break down in the tower, forming deposits of sulphur which must be removed from time to time. The spent water runs from the bottom of the still into a tank containing pipe coils, through which flows fresh ammonia water on its way to the still, thus heating the incoming ammonia-water.

The ammonia gas, from the still, is passed into sulphuric acid, much of which is recovered from the acid-tars of the refinery, as has been noted (page 84). The vessels in which the sulphate is formed are large lead-lined tubs or saturators, or "cracker boxes". The gas bubbles into the acid through holes in a lead coil in the bottom of the vessel, and sulphuric acid constantly flows into the box. In the older plants, the ammonium sulphate thus formed: $2NH_3+H_2SO_4=(NH_4)_2\ SO_4$, falls into a well at the center of the bottom of the vessel, and is lifted from it into cars with perforated bottoms by a steam ejector. The excess acid is drained from the cars, falls onto a lead-lined table, and flows back to the saturators. The sulphate in the cars is then sent to storage bins, and, after standing a while to dry, is ready

96 Williams, G. C., On the presence of pyridine in naphtha from the bituminous shale at Dorsetshire: Phil. Mag., 4th ser., vol. 8, 1854, pp. 209-212.

Williams, G. C., On the volatile bases produced by the destructive distillation of the bituminous shale of Dorsetshire: Jour. Chem. Soc., vol. 7, 1854, pp. 97-107.

Robinson, G. C., and Goodwin, W. L., New bases of the leucoline series: Trans. Roy. Soc. Edinburgh, vol. 28, 1879, p. 561; vol.. 29, 1880, pp. 265 and 273.

Garrett, T. C., and Smythe, J. A., The bases contained in Scottish shale-oil: Jour. Chem. Soc., vol. 81, 1902, pp. 449-456; vol. 83, 1903, p. 763.

97 Conacher, H. R. J. Personal communication, 1920.

for the market. More modern practice evaporates the sulphate solution to saturation in vacuum-pans, and the precipitated salt is dried in a centrifuge, and all traces of adherent acid neutralized.

The acid recovered from the refinery is run into separate saturators, and ammonia passed into it until the saturation point has been reached. The solution is then drawn from the saturators, run to settling tanks for the removal of tar, and then run into the continuous saturators, where it serves to dilute the incoming fresh acid to the proper point, or is also concentrated to saturation in vacuum-pans, and dried in centrifuges.

The exhaust steam and waste gases from the saturators are passed to the retorts, and utilized for the formation of ammonia from the shale, and the spent water from the ammonia stills is pumped to the spent-shale dump and allowed to filter through it, before being allowed to escape from the works.

Most works purchase their acid, but two of the companies have their own acid plants.

The ammonium sulphate produced by the older method, though not perfectly dry, commands a ready market. As mentioned above, in the most modern plants the sulphate is concentrated in vacuum-pans, washed, and finished in centrifuges, and is of very good quality.

The ammonium sulphate is sold and rated on a basis of 25 per cent ammonia content. It requires about 1850 pounds of sulphuric acid (sp. gr. 1.72) to make a ton (2,000 pounds) of ammonium sulphate. About 50 to 60 per cent of this acid is recovered from the refinery acid-tars.

COST OF INSTALLATION OF AN OIL-SHALE PLANT IN SCOTLAND.

It is difficult to estimate, with any degree of accuracy, the cost of an oil-shale retorting and refining plant at the present time, because of abnormal conditions and the fact that complete plants have not been erected in Scotland for over 15 years. On the authority of operators in that country, however, a retorting plant, including shale breaker, tips, retorts, condensers, tankage for crude products, haulage for spent shale, ammonium-sulphate works and a part of the power plant, would cost, in Scotland, under conditions, prices, and exchange rates prevailing in 1919, at least $5,000 per retort. As it seems that a retort should be rated by its oil output per day, rather than shale through-put (p. 73), it may be said that the investment cost of a Scotch retorting plant, complete as described above, is $5,000 per 96 gallons of oil per day (it is not safe to consider that the average oil production of a Scotch retort, under present methods of treating Scotch shales, is over 96 gallons per day). Retorting plant investment cost is therefore approximately $52.08 per gallon

oil per day, or about $2,190 per barrel (42 gallons) of oil per day. This is much less than the average "well-cost" per barrel of petroleum, but in the latter case production costs are much lower than the cost of producing an equivalent quantity of shale-oil.

Therefore the writer calculates that a Scotch oil-shale retorting plant, capable of receiving shale from the mine, producing from it 1,000 barrels of crude oil and a corresponding quantity of finished ammonium sulphate, per day of 24 hours, and disposing of the spent shale, would cost approximately $2,190,000. A refinery capable of completely refining 1,000 barrels of crude petroleum per day would cost about $1,000,000; a complete shale-oil refinery probably more. Thus a complete plant of the Scotch type capable of treating 1,000 tons of 42-gallon oil-shale per day, and making a complete line of finished products, including ammonium sulphate, would cost about $3,190,000. This figure does not include cost of land for plant site, mine, mine equipment, opening up mine, land for spent-shale dump, camp for workers, and the like. It does include complete retorting and refining equipment, ammonium-sulphate works, and auxiliary buildings and equipment.

MARKETING.

Oil-shale companies in Scotland market their products in a manner somewhat similar to that of the petroleum industry of the United States. Most shale-oil production is marketed in the British Isles, particularly in Scotland. The oil-shale companies, and many marketing concerns and large consumers, own their own fleet of tank-cars. The cars are, however, much smaller than those commonly used in the United States. A part of the paraffin wax is exported, and on account of its high purity, is in good demand.

SUCCESS OF THE SCOTCH OIL-SHALE INDUSTRY.

All of the oil-shale companies in Scotland have not been commercial successes. In 1871 there were 51 companies engaged in the industry, but in 1919 there were only six, and in 1920 these were consolidated. The successful companies have been those that, efficiently organized and well financed, have been able to employ the best technical skill, utilize labor-saving devices, and operate on a large scale. The industry has been favored by several factors, such as the relatively low cost of labor, large yields of ammonium sulphate, high cost of competing petroleum products, and situation in a densely-populated industrial region (near Glasgow and Edinburgh), where supplies and a market were close at hand.

From 1871 to 1916, the production of shale-oil increased from 593,310 to 1,965,000 barrels, whereas the production of ammonium sulphate in the same period rose from 2,350 to 59,400 long tons. This indicates that increasing effort has been directed towards the recovery of ammonium sulphate. Undoubtedly the increased recovery of this product has been a large factor in the success of the industry in Scotland.[98]

In spite of the favorable factors, which undoubtedly aided in the success of the Scotch oil-shale industry, even the larger companies have had a hard struggle to compete with petroleum products imported from Russia, Roumania, Mexico, Dutch East Indies, and the United States. Retorts were improved to produce a better oil and a higher yield of ammonia. Mechanical labor-saving devices were invented, developed, and installed, the refinery operations cheapened and improved, and economies of many kinds introduced. The chemicals used in refining were recovered; and the recovered tars, the removal of which had involved expense, became a source of profit as fuel.[99]

Even now the struggle for existence continues, especially as the lessened price for ammonium sulphate, because of its growing production from by-product coking plants, gas producers, etc., has increased the difficulties of the operators. Labor costs have increased also. In 1919, when the shale miners struck for an increased pay equivalent to the scale awarded the coal miners in Great Britain, the companies suspended operations of some of the retorts until the miners returned to the same scale. Later, the shale companies conceded the new terms voluntarily. Finally, in 1919, the existing companies consolidated, as a subsidiary of the Anglo-Persian Oil Co., in the hopes that a centralized and more efficient organization would permit more economical operation. Also, arrangements were made to have the surplus refining capacity of the shale works provided with crude petroleum, imported by the Anglo-Persian Oil Co., which again would assist in economic operation and reduction in overhead costs.

According to reports[100], in 1920 the amalgamation was expected to remove, for the present at least, all danger of failure of the Scotch industry. It has been possible under the consolidation to decrease the miner's time of employment, from an 8 to a 7-hour day, and to continue the old scale of wages. Further economies are to be effected and even now it is too soon to appreciate the full beneficial results of the reorganization.

An interim dividend of 7 per cent, less income tax, for the six months ending October 31, 1919, and a similar dividend for the

98 Gavin, M. J., Hill, H. H., and Perdew, W. E., Notes on the oil-shale industry with particular reference to the Rocky Mountain District: Bureau of Mines Reports of Investigations, Serial No. 2256, April, 1921, p. 5.

99 Steuart, D. R., The oil-shales of the Lothians, Part III, The chemistry of the oil-shales: Mem. Geol. Survey Scotland, 1912, p. 137.

100 Petroleum World, vol. 17, No. 238, July, 1920, pp. 301-302.

five months ending March 31, 1920, was ordered paid on preferred stock of the reorganized company. Likewise a dividend of 7 per cent, less tax, for the 11-month period ending March, 1920, was declared on the common stock.

Early in 1921 the efforts of the reorganization had apparently much reduced the amount of shale being mined. The Scotch refineries were reported as operating to capacity, partly on shale-oil and partly on imported Persian petroleum.

Table 20 summarizes the financial statements of the four leading companies for the fiscal years 1916-17, 1917-18, and 1918-19.

Points of note in the preceding table are the small capitalization, fair profits and dividends, and strong working capital. No plants have been erected or materially enlarged within the past 15 years, and repairs in that time have probably been routine and not particularly expensive.

DISTRIBUTION OF COSTS IN THE SCOTCH OIL-SHALE INDUSTRY.

Table 21 shows the percentage of costs attributable (in 1919) to different items entering into the total cost of producing refined shale products. The distribution as applied to retorting does not include interest, depreciation, or certain other fixed charges, which, considering the investment represented by the retorts, amounts to a considerable proportion of the total cost of retorting.

TABLE 21—Percentage distribution of costs in the Scotch oil-shale industry—1919.

Mining. a	Per cent of total mining costs
Miners' wages, measurements, and extras	36.67
On cost (below) (dead work expenses)	13.33
On cost (above) (dead work expenses)	7.50
Coal	7.50
Stores, including rails, castings and wire rope	10.00
Ties and mine timbers	8.33
Royalties (lordship)	3.33
Right-of-way	1.67
Overhead, general, and fixed charges	11.67
Total	100.00

Retorting. b

Includes taking shale from where it is delivered from mine to the spent-shale "bing", including making crude oil, crude scrubber naphtha, and ammoniacal liquor.

	Per cent of total retorting cost
Total labor (wages)	42.86
Total stores, including coal for producer gas and steam	57.14
Total c	100.00

a Pre-war costs of mining were 45 per cent of 1919 costs.
b Pre-war retorting costs were 43 per cent of 1919 costs.
c This distribution does not include overhead, general, and fixed charges, which the writer estimates would be at least 20 per cent of the total retorting cost.

Refining. d

	Per cent of total refining cost
Coal	26.67
Chemicals	33.33
Wages (labor)	20.00
Stores	3.33
Total fixed, general, and overhead charges	16.67
Total	100.00

Ammonium sulphate production.

	Per cent of total production cost
Wages (labor)	11.67
Stores, including lime	5.00
Sulphuric acid	60.00
Coal, general, fixed, and overhead charges	23.33
Total	100.00

d Pre-war costs of refining were 43 per cent of 1919 costs.

Table 22 shows the quantity of products finished from one ton of shale of the quality being worked in 1919, and Table 23 the percentage distribution of the total cost of producing from one ton of shale the finished products shown in Table 22. The distribution shown in Table 23 does not include fixed, general, and overhead charges on retorting, but does include these items in the costs of the other operations.

TABLE 22—Quantity of products finished from one ton of Scotch oil-shale in 1919.

Product	Quantity	Per cent of crude
Naphtha (450° F. end point), gallons	2.45	10
Burning oils, gallons	6.13	25
Gas and fuel oils, gallons	5.88	24
Lubricating oils, gallons	1.72	7
Wax, pounds	17.70	10
Coke, pounds	3.62	2
Ammonium sulphate, pounds	35.70	

TABLE 23—Percentage distribution of costs of making finished products from one ton of Scotch oil-shale in 1919.

Operation	Per cent of total cost
Mining	52.97
Retorting *a*	18.56
Refining	15.10
Production ammonium sulphate	13.57
Total	100.00

a Does not include fixed, general, and overhead costs of retorting.

APPLICATION OF THE SCOTCH INDUSTRY TO AN AMERICAN OIL-SHALE INDUSTRY.

Excepting the plants of Australia, which receive a government bounty on oil produced, and the small scale operations in France, the only commercial oil-shale industry in the world at present is that of Scotland. That country has been treating oil-shales in a commercial way for over 60 years. The Scotch retort, and retorting and refining procedure, have been undergoing improvements for over 50 years, and have changed little in the past 15 years. This evolution took place under extremely practical conditions, especially as the shale operators were under the pressure of severe foreign competition. It is evident, therefore, that the present retort is the best apparatus the Scotch operators know of in which to treat their particular shale under their particular conditions. It seems equally evident that the United States in which an oil-shale industry is just beginning, should not fail to take advantage of the knowledge gained during the many years of Scotch practice, and should endeavor to avoid the mistakes made in the early stages of the industry in Scotland. This is said with full realization that shales and conditions in the two countries are dissimilar. Scotch knowledge and practice should be applied and tried in this country as far as they can be applied successfully, and from this stage, our own experience should guide us.

The possible use of Scotch practice in this country is discussed on pages 119 to 127, but it may be well to indicate here some lessons that may be learned from a study of the Scotch oil-shale industry and applied in part, at least, to the potential industry of this country.

From a technical standpoint, we find that—

(1) The character of the crude oil produced, and thereby the nature, quality, and quantity of its products is influenced greatly by the conditions under which it is produced.

(2) Inasmuch as the margin of profit in oil-shale operations is small, every possible technical economy must be introduced into the plants.

(3) The refining operation must be suited to the nature of the oil; petroleum refining methods probably will not apply entirely, or well, to shale-oil refining.

From the standpoint of business practice, it is particularly evident that oil-shale operations require:

(1) The best kind of executive and technical ability; (2) a strong, economical, efficient, and highly organized control; (3) a large amount of capital, upon which only a nominal yield can be expected; and (4) large scale operations, conducted with the utmost regard for economic conditions and prevention of waste.

HISTORY AND PRESENT STATUS OF THE OIL-SHALE INDUSTRY IN THE UNITED STATES.

In 1860 there were 53 companies, mostly in the eastern part of the country, producing oil by the distillation of various kinds of bituminous substances.[101] Many of the companies operated under licenses from the Young Company of Scotland. The methods used were crude, and the materials treated ranged from bituminous and cannel coals to some true oil-shales. The desired product was kerosene or ''coal oil'', which latter term survives since the time when most of the kerosene used in this country was really derived from coal and the like. Some of these companies were only just getting started when the American petroleum industry came into being, reducing the price of kerosene to such an extent that ''shale-oil'' operations became unprofitable, and the plants were abandoned, or adapted for use in refining petroleum.

In 1914, Woodruff and Day[102] published the result of an investigation of the oil-shales of the Green River formation in Colorado. That a mineral, from which oil could be obtained by destructive distillation, existed in parts of Colorado, Utah, and Wyoming, was well known; but this report, followed by those of Winchester[103], which have been widely quoted, gave rise to a remarkable interest in oil-shales in the United States during 1916, and the beginnings of an attempted development of an oil-shale industry in this country. Since that year an increasing amount of work has been done on oil-shale, but up to the time of this writing (March, 1922), no commercial oil-shale operations have been started in this country, and the industry does not yet exist, except in the literature of promotion organizations.

Many retorts of various designs have been proposed, several patented, and a few have been erected, but most of these have been so small that they must be considered as experimental or demonstration plants. Many investigators and organizers are working in good faith, but the potential industry has suffered much harm by the fake promoter and his promotion companies. It is reasonable to say that over 100 companies have been organ-

101 Bacon, R. F., and Hamor, W. A., The American petroleum industry: Vol. 1, pp. 203-212.
Baskerville, C. E., Economic possibilities of American oil-shales: Eng. and Min. Jour., Vol. 88, 1909, pp. 149-154.

102 Woodruff, E. C., and Day, D. T., Oil-shale in northerwestern Colorado and northeastern Utah: U. S. Geol. Survey, Bull. 581, 1914, pp. 1-21.

103 Winchester, D. E., Oil-shale in northwestern Colorado and adjacent areas: U. S. Geol. Survey, Bull. 641, 1917, pp. 139-198.
......................., Oil-shale of the Uinta Basin, northeastern Utah, and results of dry distillation of miscellaneous shale samples: U. S. Geol. Survey, Bull, 691, 1918, pp. 27-55.

ized, ostensibly for the purpose of developing oil-shale in some way or another, but really for the purpose of fattening the pocketbooks of the promoters.

It is not difficult to see the reason for this situation. The country, in the years 1918-1920, was in the height of a speculative fever. Many had, during the war, made their first investments in the purchase of Government Bonds and the like, and not having learned to differentiate between an investment and a speculation, were easily convinced that their present investments should be changed into ones yielding a higher return; nor did they know the ear marks of a fake industrial stock. This speculative fever seems to be abating at the present time; nevertheless, the country is literally "wild over oil". In the popular imagination, anything connected with oil is a source of immediate wealth; and stock promoters dealing in oil-shale stock do not hesitate to make the prospect more dazzling, by presenting all sorts of impossible estimates of assured profits in oil-shale operations.

Such tactics can not but hinder the development of legitimate oil-shale operations. To those who are sincerely assisting the development of an oil-shale industry by laboratory research and by the attempted development of well-founded processes, well knowing that the chances at present are not strongly in favor of making an immediate return on the investment, much credit is due. These are the ones who are really building a firm foundation for an oil-shale industry in this country.

TENDENCY OF RETORT DESIGN IN THE UNITED STATES.

In the neighborhood of 75 proposed processes for the treatment of oil-shale have come to the attention of the writer during the past three years. Small plants, mostly for experimental and demonstrative work, have been erected in various parts of the country. Two or three are in Hoboken, N. J.; one or more in Illinois and Kentucky; one in Kansas City; several in Denver, Colorado; a half dozen or more in the region between Denver and Salt Lake City, Utah; two or three in Salt Lake City; and at least two in both Nevada and California. Plates XVI and XVII show the size and general appearance of typical oil-shale installations in this country. None of these plants can be considered as ever having operated commercially; in fact they are not large enough to be considered commercial installations, in the sense of being able to produce shale-oil in commercial qualities and quantities.

In studying the proposed processes and plants, the tendency to get away from the Scotch type of vertical retort is noted; only two or three are similarly designed, and even these are modified, mostly in essential details.

A. VERTICAL OIL-SHALE RETORT, COLORADO

B. INCLINED RETORT PLANT, COLORADO

A. VERTICAL RETORT BATTERY, NEVADA

B. VERTICAL RETORT BATTERY, UTAH

C. VERTICAL RETORT INSTALLATION, UTAH

OBJECTIONS TO THE SCOTCH OIL-SHALE RETORT IN THE UNITED STATES.

The chief objections to the use of the Scotch retort in this country are: (1), its low through-put per unit, with consequent large initial plant cost; (2), its design, for the purpose of producing a maximum quantity of ammonia from Scotch shale; and (3), the doubt that it will treat richer American shales, based largely on the operation of a so-called modified Henderson retort, in Colorado in 1916 and 1917. These objections should not condemn the Scotch retort, at least for trial, in this country, especially when the objections seem to have little or no basis in fact.

1. The capacity of the Scotch retort seems to be low, yet the experience of the Scotch operators, working for maximum yields of both refined oils and ammonia, consistent with the highest economic return, would indicate that the shales must be heated slowly if good yields, as regards both quantity and quality of finished products, are to be obtained. In the Appendix (page 179) the effects of different conditions of retorting are discussed, so it will suffice here to note that the retorting conditions applied in Scotland are probably those that yield the greatest profit per unit of shale. Other conditions might, and probably would, yield a larger amount of crude oil; a higher recovery of finished products might be obtained; or more ammonia might be produced, but the net profit would probably be reduced. It is pertinent here to mention that highest yield of crude oil does not necessarily lead to largest production of finished products of highest quality (see page 179).

Altogether aside from the influences of slow retorting on the products, other factors are to be considered. The Scotch retort treats shale in large lumps—except for a small amount of fines well mixed with the coarse material—and therefore the rate of heating must be slower than if fine material were used. It would hardly be possible to treat fine shale in a vertical retort, because of the difficulty of removing vapors. In any retort, when finely ground shale is distilled, especially if the retorting rate is rapid, a large amount of dust passes into the condensers, and what does not "hang-up" in the condensers, passes out with the oil. This makes necessary frequent cleaning of the condensers, and removal of the dust from the oil before the oil is sent to the refinery. Even with the Scotch vertical retorts, dust troubles are not entirely avoided. Since large lumps of shale are treated, the cost of crushing is small. The cost of fine-grinding the richer American shales will probably be relatively great. These are some of the advantages of the Scotch retort that tend to offset its low capacity. (See also page 124.)

The vertical retort offers many advantages over an inclined or horizontal type, inasmuch as passage of shale through and

from the retort is easily insured by simple mechanical devices. It would seem that complicated conveying and discharge apparatus should be avoided as much as possible in retorts operating at the temperatures of, and under the conditions obtaining in, oil-shale distillation. It is much cheaper to utilize the force of gravity for conveying than that of complicated mechanical devices, prone to get out of order; and finally the slow rate of depreciation, infrequent repairs, and continued operation without shut-down certainly, in a measure, offset the high installation cost of the Scotch type of retort.

Retorts better than the Scotch type may be developed, or it may be modified, but the writer sees so many favorable features in it, coupled with successful commercial operation for many years, that he believes it worthy of serious consideration and thorough trial on the oil-shales of America.

2. The objections to the ammonia-production feature of the Scotch retorts are, (a) it is necessary to use steam in retorting, with an immense additional condensing surface for condensing this steam, and (b) the general belief is that American shales do not contain enough nitrogen to warrant the production of ammonia from them. Attention is called to the fact that steam serves many functions in the retort other than that of acting as an ammonia producer (page 48), and its use is probably essential in the production of good-quality oils from vertical retorts. As regards the nitrogen content of American shales, Table 6 (page 30) indicates that in this regard they will average well with Scotch shales. The writer does not venture to predict whether American oil-shales will be treated to recover their nitrogen as ammonia, but the point is that nitrogen is present in comparatively large quantities in oil-shales of the United States.

3. In 1916, a small retort, said to be a modification of the Scotch Henderson, was erected near DeBeque, Colorado. Except for minor details, this retort was all the modern Henderson is not. It was of cast iron with 1-inch walls, 15 feet high and tapering, 12 inches in diameter at the top and 20 inches at the bottom. A piece of 12-inch casing, 8 feet long, set vertically at the top, served as a hopper, and one toothed roller at the bottom, operated intermittently by hand, was intended as a discharge mechanism. The retort was set in a brick furnace, not at all like that used with the Henderson retort, and was heated by combustion of the gases produced in retorting, supplemented by raw shale burned in a fire box. A small exhaust pump drew vapors away from the retort. Provision was made for passing steam into the bottom of the retort.

The retort was filled with rich, massive shale, and heated. As is probably the case with many rich shales, when heated rapidly, this shale intumesced and adhered to the walls of the retort, stop-

ping operations. From the results of this one retort, the idea has arisen that Scotch retorts can not be used for rich American shales. By referring to the description of the Henderson retort (p. 69) it is evident that the retort under discussion was a crude attempt to imitate it. The test was by no means fair, and should not be considered conclusive. One of the features of the Scotch retort is the fact that the shales are heated slowly, and the products removed rapidly by the use of excess quantities of steam. As far as coking or adhering of the shales to the walls is concerned, experiments conducted by the Bureau of Mines indicate that these difficulties can be avoided with most shales if they are heated slowly and the products removed as they are formed. Later tests on the type of retort referred to above, somewhat modified, have given more favorable results, but even yet it is by no means more than a crude attempt to imitate the modern Scotch retort.

In 1920, a four-retort experimental Pumpherston unit was erected near Elko, Nevada, by a private corporation, with which the Bureau of Mines cooperated to the extent of assigning one of its consulting engineers to direct the construction and operation of the plant. Although the retorts were built in accordance with plans obtained from Scotland, the brick portions cracked and pulled apart so badly when first heated that repairs would have involved the practical demolition and reconstruction of the plant. The failure probably was due either to unsuitable materials of construction or to too rapid heating of the newly constructed retort. Scotch operators stress the necessity of using specially selected fire-clay in the manufacture of retort bricks, and in building the brick parts of the retorts. They also emphasize the danger of damage to the brick-work if the retorts are heated too rapidly when first put into operation, or after they have been down for repairs. The failure of the Elko plant, however, should not be taken as indicating the unsuitability of the Scotch retort for the treatment of American shales.

GENERAL DISCUSSION OF TYPES OF AMERICAN OIL-SHALE RETORTS.

It has been noted that the present trend in the development of oil-shale retorts in this country is away from the Scotch type. The principal features of proposed American retorts may be briefly outlined by dividing them into several general types. After these general types are described, a table (Table 24, page 108) follows, which gives the names of various processes, where the names and addresses of the inventors or owners are known to the writer. The table also classifies the processes according to the classification given below:

1. Horizontal retorts with conveyer systems for moving shale through the retorts and discharging it.

(a). Cylindrical. In this type, either one or several superimposed horizontal cylinders are provided with conveyers of various kinds, usually Archimedian screws. If one cylinder is used, shale feeds in at one end and is subjected to rising temperature on its travel through the retort, being finally discharged in a spent condition. If more than one cylinder is used, the shale passes in series through them, and is subjected to a higher temperature in each. With this type of retort vapors are often removed at several different points, on the theory that oils of different qualities are distilled from the shale at different temperatures, and that by taking advantage of this condition a fractional "eduction" will result, yielding refined or partly-refined fractions direct from the shale. There is undoubtedly some truth in the theory, but the extent of such fractionation does not appear to be sufficient to be of practical value. (See page 179.) In many cases what has seemed to be fractional "eduction" has really been fractional condensation, taking place either within the retort, or in the condensers.

Retorts of this general type were tried in Scotland early in the history of the industry (page 63) and were abandoned as being difficult to control, and thereby producing an unsatisfactory oil. This does not necessarily indicate that they will be unsatisfactory for American shales and conditions, however.

(b). Rectangular. This type usually consists of a flat hearth, heated from below, and covered with a hood or top which leads the oil vapors to the vapor mains. The shale, which is ordinarily crushed to a size that will pass a 10-mesh screen, is usually fed onto the hearth by a screw conveyer. The feed end of the hearth is generally the coolest part, and the temperature increases progressively as the discharge end is reached. The shale is

moved across the hearth and discharged from the retort by a series of rabbles, rakes, toothed conveyers, or the like. Discharge is usually into a water-seal. Provision is frequently made to secure whatever benefits there may be in the fractional "eduction" theory.

2. **Inclined retorts with conveyers or agitators.** This type of retort needs little description, as the title given is nearly self-explanatory. Of the various types proposed, the retorts are inclined at different angles to the horizontal, varying from 5 or 10 degrees to perhaps 45 degrees. In different retorts, the cross-section is circular, semi-circular, or a segment of a circle. One or two are rectangular. Various types of agitators or conveyers are used to keep the shale in motion through, and discharge it from, the retort. These vary from scrapers oscillating from side to side and scraping the bottom of the retort, to belt or bucket conveyers actually carrying the shale through, and from, the retort

3. **Continuous, externally-heated vertical retorts.**

(a). Modifications of Scotch type. At least two modifications of the Scotch vertical retort have been proposed. One is based on the Pumpherston retort, but has two separate chambers, the upper being heated by passing through it combustion gases from the lower furnace; and the lower chamber externally heated, as with the Pumpherston. The two chambers, or retorts, are connected by a duct, normally closed by a manually operated valve. In the upper chamber the shale is preheated and dehydrated by direct contact with the hot combustion gases. The shale is then dropped into the lower part, in which destructive distillation takes place. Steam is admitted into the lower retort, which may be of silica brick, and the discharge mechanism is similar to that used in the Pumpherston retort.

Another retort is a modification of the Henderson, and in commercial size will be of about the same dimensions as the latter, with the same type of discharge mechanism. The modification consists of perforated vapor pipes extending into, and through, the upper or iron part of the retort, the idea being to remove the distillation products as rapidly as they are formed, and to gain the supposed advantages of fractional eduction.

(b). Other continuously operating, vertical types. One inventor proposes a vertical retort made of two concentric castings, heated from an inside and outside flue. One casting is stationary and the other rotary, about a vertical axis. The outer surface of the inner casting, and the inner surface of the outer casting, are provided with rows of short horizontally-placed lugs or shelves. The shale feeds in at the top onto the lugs, from which it is scraped by the lugs on the moving cylinder, and falls to lower lugs and is finally discharged.

Other vertical types are based on the principles of the Wedge or Herreshoff furnaces for roasting ores. In these types, shale is fed onto an externally-heated horizontal hearth, on which it is moved by a series of plows or rabbles operated by a central rotating vertical shaft. The shale progressively falls from one hearth to a second, is scraped over it and discharged to a third, and so on, and finally discharged. Each successive hearth is heated to a higher temperature, and vapor lines usually lead from the level of each hearth.

Still other vertical types, continuously operating, discharge the spent shale from an externally-heated fire brick retort, into a furnace, in which the spent shale is burned. The fixed carbon of the spent shale is supposed to furnish, together with the fixed retort distillation gases, sufficient fuel to conduct the distillation in the upper distillation retort. Steam may also be admitted into the upper retort.

4. **Intermittent, externally-heated, vertical retorts.** A retort of this type consists of a vertical casting or refractory retort, closed at top and bottom by tight-fitting lids. The lower lid has, passing through it, and removable with it, a vapor line which reaches up to within a few inches of the top of the retort. This inner vapor line is perforated and vapors resulting from distillation by external heating are drawn in through the perforations, and downward out of the retort by a suction pump placed somewhere in the gas main or condensing system. This arrangement is supposed to provide better transmission of heat, and immediate removal of the products as they are formed from the zone of formation to a zone of lower temperature. This process may also be made continuous.

5. **Internally-heated retorts.** Several types of internally-heated retorts have been proposed. Most of them are vertical; some operate continuously, and others intermittently. Heating is effected by passing superheated steam or fixed distillation gases into one end of the retort, and taking them, with the products of distillation, out of the other end. The condensable products are condensed, the gases scrubbed, and the fixed gases heated and passed again into the retort. Any excess gas is used as fuel in the gas or steam superheater. In one case, a series of retorts of this type is operated, the hot gases passing into retort 1, out of 1 and into 2, and so on, as far as feasible. When the shale in retort 1 is spent, it is discharged and filled with fresh shale. Retort 2 then receives the gas first and retort 1 last.

6. **Horizontal or inclined, rotary cylindrical retorts.** This type is usually more or less like a cement clinkering kiln, and is designed to operate continuously. It is essentially a rotating cylindrical retort, externally heated, and is usually inclined about 15

degrees from the horizontal. The retort is set in a furnace and rotated by gears driven by electric motors or steam engines. Shale is fed at the cooler higher end, passes downward through the retort, and out at the lower end. The rotation of the retort, sometimes supplemented by vanes or spiral baffles, causes the shale to move downward at a uniform rate. Provision is sometimes made for taking the vapors off at several points. A proposed type of this retort is 60 feet long and 30 inches in diameter.

7. **Miscellaneous types of retorts.** One inventor treats his shales in a digestion bath of hot oil, recovering light oils by distillation from the bath direct, and heavy oils, by removal of the bath-oil from the digestor, separating the spent shale from it, and distilling the bath-oil, which also carries the heavier products resulting from distillation of the shale. This process is designed to operate continuously, the refining of the shale-oil furnishing the bath-oil, a constant amount of which is kept in the system. Another inventor combines this principle with that of ordinary destructive distillation. In this case, a bucket conveyor carries the shale into and through the hot oil-bath; and from it the same conveyor carries the residual shale to another externally-heated vertical retort, in which ordinary destructive distillation takes place.

Other miscellaneous types of retorts are (1) of the vertical, externally-heated type, with screw conveyers for moving the shale through; (2) closed ovens in which cars containing raw shale are admitted for distillation and withdrawn when the shale is spent, the vapors being taken off through openings in the oven walls; and (3) cylindrical retorts which are externally heated and given an oscillating motion during distillation. One inventor proposes to drill vertical holes into the shale deposits and set up intercommunicating passages between holes by exploding powder in the bottoms of the holes. It is then proposed to pass superheated steam down the central hole and recover the products distilled by the steam from the others.

This discussion gives only the barest outline of the more commonly proposed types of retorts for treating oil-shales in the United States. Possibly some have been omitted, as new ones are being proposed continually. Some of the proposed retorts can be rejected by the engineer as being constructed on unsound principles or practices; and others, which appear to have favorable features, can not have their commercial value determined until they are operated in a commercial manner. As previously noted, none of the above types have been operated on a scale sufficient to give much of an idea as to their feasibility under commercial conditions, and many of them exist only on paper, or in the minds of their inventors.

TABLE 24—Oil-shale processes proposed for use in the United States. [a]

Name	Type [b]	Name of inventor or owner	Address of inventor or owner
Anderson	1b	Anderson Shale Oil Co.	160 S. Broadway, Denver, Colo.
Balcom	1b	Balcom Rite-Wa Engineering Co.	St. John, New Brunswick
Bishop	1b	Bishop, J. A.	1265 Lafayette St., Denver, Colo.
Boyle	3b	Boyle, A. M.	Reno, Nevada.
Brown	6	Brown, H. L.	265 Washington Ave., Newark, New Jersey
Bussey	3b	Bussey, C. C.	Brooklyn, N. Y.
Catlin	5	Catlin, R. M.	Franklin Furnace, N. J.
Chew	3b	Chew, L. F.	18th & Blake Sts., Denver, Colo.
Colorado Continuous	3b	Krushnic, E. L.	421 Cooper Bldg., Denver, Colo.
Crane	7	Crane, A. G.	Reno, Nevada.
Day-Heller [c]	3b	The Day Co.	414 Hobart Bldg., San Francisco, Calif.
De Brey	7	Mock, S. A.	Salt Lake City, Utah
Del Monte	1a	Prevost, C. A.	Southern Bldg., Washington, D. C.
Edwards-Parks	1a	Edwards, T. H.	2161 Larimer St., Denver, Colo.
Erickson	3a	Rainbow Petroleum Products Co.	419 Judge Bldg., Salt Lake City, Utah
Galloupe	3b	Galloupe, J. H.	323 Chamber of Commerce Bldg., Denver, Colo.
Ginet	1a	Ginet, J. H.	Temple Court Bldg., Denver, Colo.
Godfrey	7	Godfrey, H. H.	Los Angeles, Calif.
Hague	1a	Hague, S. T.	Salt Lake City, Utah
Green-Laucks	3b	American Coal By-Products Co.	Denver, Colo.
Hoover-Brown	7	Brown & Co., Thos. E.	32 Nassau St., New York City
Jenson	1a	Jenson, J. B.	823 McIntyre Bldg., Salt Lake City, Utah
Johns	2	Industrial Process Engineering Co.	Rialto Theatre Bldg., St. Louis, Mo.
Lesley	6	Lesley, R. W.	611 Pennsylvania Bldg., Philadelphia, Pa.
McCaskell	7	McCaskell, J. A.	1417 E. 1st South St., Salt Lake City, Utah
Noad	3b	Noad, J.	East Ham, Essex, England
Porter	3b	Porter Process Co.	409 Symes Bldg., Denver, Colo.
Perry	3b	Perry, W. P.	Leytonstone, London, England
Prioleau	1a	Prioleau, W. L. St. J.	London, England
Randall	6	Randall, J. W. H.	15 E. 40th St., New York, N. Y.
Robinson	3b	Robinson, T.	Anaconda, Montana
Rogers	7	Rogers, L. H.	New York, N. Y.
Ryan	7	National Oil Machinery Co.	1270 Broadway, New York, N. Y.
Scott	3a	American Engineering Association	979 Woodward Ave., Detroit, Mich.
Seaman Rotary	6	Seaman Wastewood Chemical Co.	17 Battery Place, New York, N. Y.
Simpson	5	Simpson, L.	172 O'Connor St., Ottawa, Can.
Simplex	2	Mt. Logan Oil Shale Mining and Refining Co.	De Beque, Colo.
Stalmann	3a	Stalmann, O.	319 Ness Bldg., Salt Lake City, Utah
Shreves	1a	Shreves, F. G.	Denver, Colo.
Stone	3b	Stone, H. G.	Chicago, Ill.
Straight	1b	Straight, H. R.	Adel, Iowa
Thurlow	3a	Thurlow, E. W.	26 New St., Brighton, Victoria, Australia
Wallace	4 3b	Wallace, G. W.	412 Missouri Ave., E. St. Louis, Ill.
Watson	2	Watson Shale Refining Co.	205 Ness Bldg., Salt Lake City, Utah
Wingett	3b	Troy American Pet. Co.	1214 First Natl. Bank Bldg., Denver, Colo.
Whittaker-Pritchard	5	Fuel Products Corp.	110 W. 40th St., New York, N. Y.
Young	2	Young, A. V.	De Beque, Colo.

[a] This list contains only those processes the names and addresses of the owners or inventors of which are known to the writer at the date of writing (July, 1921). Others have been proposed, but few data are available regarding them. New processes are continually being proposed and invented. Few of those mentioned in the table have reached the stage of successful demonstration.

[b] Type classification refers to that in the foregoing text.

[c] The Day Co. is the owner of various types of retorts other than that referred to.

In studying the various designs of retorts, in connection with the history of the oil-shale industry of Scotland, one is struck by the fact that the majority of the types proposed have been tried and rejected by the Scotch operators as inefficient, impractical, or were replaced by better methods. This does not necessarily condemn them for use in treating American shales, which undoubtedly are different from the Scotch shales; but it is suggested that those contemplating the erection of retorts in this country ascertain if the apparatus they propose to erect, or a type similar to it, has ever been tried elsewhere, and if possible determine the reason why it was not used.

Another important fact is that most inventors of oil-shale retorts are paying little attention to the principles of destructive distillation, and apparently have little regard for the kind of oil or other products their retort will produce. The interrelation of retorting and refining are discussed in a following section (page 119), but it is apropos to point out here that the nature of the crude oil, the degree of refining it will require, and the quality and quantity of finished products that can be produced from it, depend materially on the conditions under which the crude oil is produced. Many inventors are apparently trying to devise a retort with a large output of crude oil per unit of time and installation, and are overlooking the fact that they must produce an oil of good refining quality. (See also page 179.)

It is possible that different types of retorts, or different retorting conditions may be required to produce crude oils containing maximum quantities of different desired products. It is also possible that different shales will require different types of retorts and conditions of retorting for satisfactory treatment. For this reason, if any type of retort does not operate satisfactorily on a particular shale, it may not indicate that it will not operate satisfactorily on a shale of different character.

ACQUISITION OF OIL-SHALE DEPOSITS ON PUBLIC LANDS.

Large oil-shale deposits on public lands were, in 1921, opened for lease by the Federal Government, under the terms of an Act of Congress approved February 25, 1920, entitled, "An Act to Promote the Mining of Coal, Phosphate, Oil, Oil-Shale, Gas and Sodium on the Public Domain". This is known as the Mineral Leasing Act.

The rules and regulations governing the issuance of such leases are given in General Land Office Circular 671 entitled, "Regulations Concerning Oil-Shale Leases Authorized by the Act of February 25, 1920. (Public No. 146.)" Persons interested in this subject should address a request for this bulletin to the Commissioner of the General Land Office, Washington, D. C. If a lease is desired, application should be made to the Register

of the proper Land Office, and he will give the necessary instructions and supply the required forms.

The supervision of oil-shale mining operations is under the jurisdiction of the Bureau of Mines. A pamphlet entitled, "Operating Regulations to Govern the Methods of Mining Oil-Shale, Phosphate, Sodium and Potash, Under the Acts Approved February 25, 1920 (41 Stat. 437), and October 2, 1917 (40 Stat. 297), and the Safety and Welfare of the Employees in Connection Therewith", has been issued by the bureau for the benefit of those interested and may be had upon application to the Director, Bureau of Mines, Washington, D. C., or to the Register of the local Land Office.

Because of the many points of law involved, as well as conflicting claims, it is impossible to give definite rules to pursue in obtaining oil-shale leases, and persons interested in leasing and operating deposits on the Public Domain should submit their cases to the Register of the local Land Office for individual consideration.

ACQUISITION OF OIL-SHALE LAND BY LOCATION.

Prior to the Leasing Act of 1920, oil-shale locations were made under the placer regulations. The Land Office has ruled that where such locations were in all respects legal and regular, they might go to patent, as in the case of ordinary mining claims. A few patents have already been granted to oil-shale locators. Other patents may be expected to be issued to those who have fully complied with the law and located their claims before the approval of the Act of February 25, 1920. Certain shale lands are of course already owned in fee simple, and therefore it may be possible to acquire deposits from their legal owners.

Considerable caution should be used in purchasing oil-shale lands from alleged owners, as the titles are often not at all clear, and many are now in dispute. Frequently, land surveys in the shale districts have been only preliminary, and later surveys may be expected to change some boundaries. It is reported that there have been many cases of claim jumping and fraudulent entry in claiming oil-shale lands, particularly in Utah and Colorado. Therefore, the prospective purchaser of such land is advised to take particular care to ascertain that the title to the land is clear before he purchases. Purchasers are also warned not to deal with companies who claim they can secure land under a special agreement or arrangement with the United States Government, as no such terms exist or would be allowed.

THE ECONOMIC IMPORTANCE OF OIL-SHALES TO THE UNITED STATES.

The great economic importance of oil-shales is that when the industry is properly developed, the United States will have a new domestic supply of mineral oils, which cannot be cut off in time of war, whether military or economic. Until the oil-shale resources are developed, the comforting assurance may be had that here is the raw material for a new supply of mineral oils, which come what may, will always be ready to help meet the nation's demands for oil, no matter what the world's petroleum situation may be. As so many have expressed it, our oil-shale deposits constitute a rear line of economic defense in assuring the United States a home supply of mineral oils.

The oil-shales are of great economic importance and interest to the states in which they occur. Mention is made (page 131) of the supply of miners and other laborers necessary to develop and operate a great oil-shale industry in this country. Most of these laborers will be brought into the shale districts from remote regions, will live in the shale fields, and with their families, earn and spend their money in the same locality. The enormous capital to be invested in the shale industry will add to the wealth and taxable properties of the states in which the money is invested. Auxiliary industries will develop, which again will add to the population, prosperity and wealth of the shale regions. Transportation must be furnished to the shale districts now not adequately supplied in that respect, and this should help also to open up new mining and agricultural districts. Oil products may be relatively cheaper in the regions close to the shale districts than in other parts of the country. The availability of cheap fuels near the shale districts will do much to develop unused agricultural districts of great potentiality, which occur close to the shale fields of the West.

With the possible exception of certain products, it is probable that shale-oil will not, for a long time, enter into the country's foreign trade. It would appear that all the shale-oil that can be produced in this country for a great many years will be consumed within the borders of the United States.

PROBLEMS OF AN AMERICAN OIL-SHALE INDUSTRY.

The oil-shale industry of the United States is still in an experimental stage, and it is to be regretted that much of the experimental work is not being conducted with regard to the problems that will have to be solved in commercial work. Thus we see much effort to utilize the spent shale and to work up by-products, long before the problems of the production of a satisfactory oil, and the refining of that oil have been worked out. The production and refining of shale-oil is a chemical manufacturing operation, and should be directed and controlled by experts. It is a fact, at the present time, that the majority of those working on oil-shale have little or no knowledge of the technique and technical problems presented by the substances they handle, and their possible products. The oil-shale industry is one to which much technical knowledge and skill will have to be applied before it can become a successful commercial enterprise of first importance.

MINING.

In an earlier section (Table 23, page 96) a summary of the relative costs of the different stages of producing refined products from oil-shale in Scotland was presented. The cost of mining the shale was more than half (53 per cent) the total cost of producing refined products. It is to be expected that with American shales and under local conditions, costs may differ, and the same relative distribution not apply. The figures from Scotland, however, should be of interest as a basis for cost considerations, and should, in a way, indicate where the greatest efforts towards reducing costs should be made.

Many of our shales may be worked more readily than Scotch shales, but clearly the cost of mining will make up a large part of the total cost of producing and refining shale-oil. Mining methods and the possible reduction of mining costs should, therefore, be given serious and thorough consideration.[104] In all probability a saving of 10 per cent in the cost of mining will be much more important than one of 10 per cent in retorting and refining.

Oil-shale is tougher than coal; most of the deposits are horizontal or nearly so; and those in the Rocky Mountain region usually outcrop high up in the walls of canons, and lie at altitudes of from 5,000 to 8,000 feet. In such cases, most mining engineers

104 Gavin, M. J., The next mining problem: Proc. Am. Min. Cong., Vol. 24, 1921, pp. 497-510.

believe that oil-shale will be mined by methods similar to those used in coal mining, and at about a similar cost. The impression has been created that shale can be mined by open-cut or quarrying methods, permitting the use of steam-shovels. While this is undoubtedly true in the case of some shales in the Eastern States, there are probably few deposits in Colorado, Utah, or Wyoming where steam-shovel methods can be employed. In the latter region the workable seams are, as a rule, relatively thin, and are usually covered by comparatively great thicknesses of overburden. It is now considered that most of these oil-shales will be mined by underground methods similar to those used in mining coal.

However, in many cases there are few accurate data at hand regarding the thickness and richness of the seams. Comprehensive core-drilling, or some other method of determining the thickness of the various shale seams, and of obtaining accurate samples of the geologic column in the shale formations, will do much to determine the feasibility of various methods of mining. It is recommended that work of this nature be done as soon as possible, preferably in some manner whereby the results may be made public for the benefit of all who are interested in the development of an oil-shale industry. As this manuscript is being written (1921), core-drilling is being started by three companies in the shale district, near DeBeque, Colo.

Plans for most contemplated workings in the western shale fields consider that the shale will be mined with tunnels entering from the outcrop. In such cases the retorting plant will be on the hill-side between mine-entry and valley floor, or on the valley floor. Consideration has also been given to the possibility of placing the retorting plants on the tops of the mesas or plateaus, and mining the shale by shafts from the mesa top. Shaft-mining may often be cheaper than tunnel-mining, especially when mining is being conducted at considerable distance from the outcrop. In this case the average underground haul to a centrally-located shaft will often be so much less than that with a tunnel entry that it will pay to install expensive hoists.

It is not probable that many western mines will be shaft operated, at least not until most of the shale near the outcrops has been mined. Although the tops of the mesas provide good sites for works and towns, and ample space for dumping spent shale, which the valleys and hill-sides do not always furnish, these advantages are offset by the probable difficulty of securing water supplies on the mesa-tops, the severe winter climate and heavy accumulation of snow, and the difficulty of supplying transportation.

In many of the Eastern States, the black oil-shales overlie coal seams, and are removed with the coal in open-pit or stripping operations. In such cases, or in similar cases where the shale

lies close to the surface, it is probable that steam-shovel operations will be feasible. Most of the black shales of the Eastern States are not very rich in point of oil production, and it is likely that reduced mining costs will be largely offset by the necessity of mining more shale to get a yield of oil equivalent to that produced from the richer shales of the Rocky Mountain district.

It is expected that the shales will furnish good walls, and that the problem of supports will be no more difficult to solve than in coal mining. However, in this regard it may be well to quote from an Australian publication[105] on the mining of some of the rich oil-shales in the State of New South Wales, as it is possible that similar conditions may be met with in shale mining in the United States:

"Both 'longwall' and 'pillar-and-stall' systems of extraction have been adopted; in the latter the pillars are subsequently removed. In the present working mines—Genowlan and New Hartley—the workings are on the 'longwall' system. Here explosives are unnecessary for breaking down the seam; on the contrary, great danger is experienced from the continuous cracking and shooting of the kerosene shale as the roof settles on the face. The unyielding nature of the top-holing, which consists of a band of hard cannel forming the top of the shale seam, affords no relief to the vertical pressure. Where soft bituminous coal forms the roof, it gives or crushes under stress, and thus to some extent relieves the enormous pressure of the superincumbent Coal Measures and overlying Hawkesbury series on the tough, semi-elastic kerosene shale. Where lateral expansion is rendered possible by open spaces—i. e., working-faces—the tension is so great that cracking and shooting are almost continuous as settling proceeds. Further release by holing is therefore attended with considerable risks to the miners, who are compelled to work behind breast-boards, and cover their eyes with wire-gauze guards as protection against flying fragments of keen-edged shale. The tendency of kerosene shale to conchoidal fracturing under stress at right angles to the stratification increases the development of sharp edges which cut like knives. Numerous accidents have resulted, some unfortunately attended with serious impairment of sight.

"Similar phenomena have been noted by Mr. E. C. Andrews, Geological Surveyor, in connection with the 'kicking' or 'spitting' shale of the Baker's Creek Proprietary Gold Mine, at Hillgrove, where an unfortunate miner was cut in two by a large flying fragment which had previously passed through a 3-inch x 2-inch scantling. The slightest disturbance of the apparent equilibrium between compression and expansion, caused by drill or hammer, in this instance also caused sharp-edged conchoidal fragments to fly off with extreme violence.

"At Joadja, where the overburden is greatly less, and where also in the early workings soft bituminous coal formed both top and bottom holing, the tension of the face was so slight that no trouble was experienced from 'shooting'. In the present workings under heavier cover, with the pressure unrelieved by a yielding coal medium on roof or floor, a tendency to 'shoot' is being manifested. Explosives (gelignite) are used for breaking-down, as it is also used at Genowlan, together with compressed powder, for 'brushing' or deepening the roadways, the latter being used in the New Hartley workings.

"None of the kerosene shale workings have been troubled with firedamp, naked light always being used, in which paraffin wax or heavy oil is burned. Scotch shale-mining is similarly free from natural gases.

"Ventilation in all cases is by air-shafts and furnaces."

105 Carne, J. E., The kerosene shale deposits of New South Wales: Memo. Geol. Survey New South Wales, 1903, pp. 84-85.

MINE GASES AND THE EXPLOSIBILITY OF OIL-SHALE DUST.

In an earlier section (p. 61), it has been noted that the Scotch miners have not been troubled with explosive or poisonous gases in the shale workings, and that the dust produced in Scotch shale-mining operations is not inflammable or explosive. This must not be taken to determine positively that similar conditions will obtain in American shale mines.

As regards explosive gases, rich American oil-shales give off a strong odor like petroleum when freshly broken, and the writer has been advised of one shale mine where, after each shot is fired, gas, smelling much like natural gas, is given off copiously, and can be detected at a short distance from the mine entry for some time after the shot.[106]

There is a possibility that oil-shale dust may present a source of danger in American mines, particularly where comparatively rich material is being worked. At its Pittsburgh Experiment Station the Bureau of Mines has examined one sample of Colorado oil-shale to determine its explosibility, and the report on the test is given in full below, as it will undoubtedly be of interest to all those who are mining or contemplating mining oil-shale in this country. The shale tested was not assayed to determine its richness, but from the proximate analysis given the writer estimates that it will yield about 35 gallons of oil to the ton, and therefore may be considered a fairly representative type of Colorado shale. Similar tests will be made on shales from this and other districts as soon as possible, to determine whether the richness and mineral composition of an oil shale have important bearing on its explosibility.

THE EXPLOSIBILITY OF OIL-SHALES.[107]

"The material tested was a 20-pound sample of Colorado oil-shale sent to Mr. J. W. Paul, Chief of coal mining investigations, by Mr. Daniel Harrington, District Engineer of the Bureau, to whom the sample was given by the Colorado Commissioner of Mines. The material was crushed, and a kilogram portion taken, which was pulverized to approximately 20 mesh. The analysis of a sample of this pulverized dust follows:

	As received Per cent.	Moisture free Per cent.	Ash free Per cent.
Moisture	0.35	....	....
Volatile and fixed carbon	37.37	37.40	100.00
Ash	62.38	62.60	....
	100.00	100.00	100.00

"**Experimental Work.** For testing the explosibility of dusts less inflammable than coal dust, there is in use in the Pittsburgh laboratories a horizontal dust explosion gallery. This apparatus is of wood, 14 feet long and 6 inches square, inside dimensions. At intervals of one foot

106 Jensen, J. B., personal communication, 1920.

107 Leighton, Alan, and Lentz, H. E., The explosibility of oil-shale: Unpublished report of the Pittsburgh experiment station of the Bureau of Mines, 1920.

along the floor of the gallery are arranged jets, from which dust may be projected as a cloud into the gallery upon the firing of a small charge of powder in a so-called dust-raising cannon.

"The rear of the gallery is closed with a tight-fitting breech-block, immediately in front of which is placed a small cannon containing a hole one-half inch in diameter and two inches deep. Into this cannon are loaded five grams of FFF-G black rifle powder, and immediately in front of the cannon are placed 10 grams of the same powder. Ignition is obtained by means of an electric igniter, which is placed in such a way that the flame lights the outer end of the powder charge in the cannon. By this arrangement a hot powder flame is produced without concussion. After the gallery is loaded with dust, the two cannons are fired simultaneously, and if ignition is to take place, flames will issue from the end of the gallery.

"This gallery has been standardized with Pittsburgh coal dust against tests conducted at the Experimental Mine. To illustrate: In the Experimental Mine it has been found that the flame from a blown-out shot of four pounds of black blasting powder will just ignite a mixture of pulverized Pittsburgh coal dust, 40 per cent, and pulverized Experimental Mine shale, 60 per cent. Such a mixture in this gallery ignites and flame is propagated 14 feet to the end. The flame produced in a blank test when the gallery is loaded with shale dust is from 8 to 9 feet. This is taken as the standard of comparison. On the other hand, the flame from a mixture, containing 70 per cent shale dust and 30 per cent coal dust, extends 11 feet in the gallery, as does the powder flame when the gallery is empty. This is a mixture that will just propagate an explosion in the mine if an explosion has been started. Similar figures are obtained in the gallery when coarser dusts are used.

"The pulverized oil-shale was tested in this manner, with the result that the flame length was 11 feet. This is the flame produced by a mixture of 70 per cent pulverized shale and 30 per cent pulverized Pittsburgh coal dust, a mixture which will not be ignited by the standard blown-out shot at the Experimental Mine, but which will propagate an explosion if one is started in some other medium.

"Following this test, experiments for comparison were conducted in the Clement-Frazer apparatus. This apparatus is described fully in U. S. Bureau of Mines Technical Paper No. 141, and consists essentially of a small electrically heated coil suspended within a glass globe of definite dimensions. A small dust cloud is blown across the heated coil, and the pressure generated within the globe is taken as an indication of the inflammability of the dust. Following the usual procedure, three milligram samples of the shale dust were projected against the coil, the temperature of the coil being 1200°C. Ignition resulted, the average pressure produced being five pounds. This is the pressure that would be produced by a mixture of pulverized Pittsburgh coal dust 35 per cent, and pulverized shale dust 65 per cent. It is to be noted that this result closely checks the result obtained in the horizontal gallery.

"Following this, a cloud of dust was blown through a Bunsen burner flame. While the particles glowed as they passed through the flame, there was no evidence that flame was propagated through the dust cloud.

"It having been determined that the solid material could be heated to a point where distillation took place with sufficient rapidity to support flame combustion, portions of the dust cloud were blown against a small hot incinerating dish. This dish was placed in a small muffle furnace, and, as the temperature of the furnace was raised each 50 degrees, the dish was quickly withdrawn and the cloud caused to pass over it. As soon as the dish reached 800° C., the dust particles that fell onto the dish flamed up, but there was, at no time, an indication of a general ignition of the dust cloud up to a temperature of 1000° C.; the highest reached.

"**CONCLUSIONS:** These experiments indicate that the oil-shale cannot be considered as a highly inflammable dust. The dust will probably not be ignited by the flame from an ordinary blown-out shot, but is fully capable of propagating an explosion once started".

It is recommended that miners, working in rich oil-shale, take the same precautions that they would when working a gassy or dusty coal-seam, until such time as they are certain that no dangerous conditions in the way of gas and explosive dusts are to be encountered.

The State of Colorado has already provided that the mining of oil-shale will come under the jurisdiction of the State Bureau of Mines. At first, at least, it would appear that the State Bureau will require the same precautions to be taken in shale mining as are observed in mining coal. The following[108] indicates the attitude of the Colorado Bureau, and also gives further information as to the possible hazards to be encountered in oil-shale mining:

MINING REGULATIONS.

"The attention of all shale mine operators in Colorado is called to the fact that, under the existing laws a shale mine, like all mines and quarries, except coal mines, comes under the jurisdiction of the [State] Bureau of Mines; also a shale-retorting plant is a metallurgical plant and is under the same jurisdiction. It is the duty of the Commissioner of Mines to make such rules and regulations as are necessary, in addition to the statutes, to reduce the hazards of mining and metallurgical operations as far as circumstances permit and to safeguard in every possible way the lives and health of the miners and other workers. The mine inspectors are to see that the laws, rules, and regulations are observed and to make such recommendations as may be necessary to carry out the spirit of the law.

"Any person or corporation starting operations is required by law to notify the Bureau of Mines so that the inspectors may not overlook any operating properties.

"At this time it appears probable that the first shale mining on a commercial scale will be underground, using the same methods as in mining coal. Consequently the same hazards will be encountered and the same precautions must be observed as in coal mining. Open-cut mining or quarrying must be conducted under the same regulations as are observed in other quarries.

"In underground shale mines there is a possibility that inflammable gas will be encountered. There does not appear to be enough gas to be dangerous in the shale itself. There is, however, a considerable amount of inflammable gas in the underlying strata, as shown by numerous gas wells at DeBeque and elsewhere. It is quite possible that this gas may find its way into the shale through cracks or minute fissures in the underlying rocks. If, in the course of mining, one of these fissures is tapped, the gas, being under pressure, will escape into the mine, forming an explosive mixture with the air, and if it comes in contact with an open flame or spark, an explosion will result. To guard against this it will be necessary, after shots are fired, to have the mine inspected by a qualified fire boss before the employees are allowed to enter it.

"Another source of danger, and one that is certain to be present, is the dust. Mining operations of any sort are conducive to the formation of large quantities of fine dust, which collects on the floors and irregularities of the walls of the workings. Shale dust is highly inflammable[109] and like coal dust, flour dust, or the dust of any other combustible substance, will, under certain conditions, form a dangerously explosive mixture with air. This explosive mixture may be ignited by the open flame

108 Lunt, H. F., Dalrymple, J., and Duce, J., The oil-shales of northwestern Colorado: Colorado State Bureau of Mines Bull. No. 8, 1919, pp. 45-46.

109 The inflammability of shale dust may be shown by letting a handful of it trickle through a hot flame. The particles will ignite and give the effect of a miniature roman candle.

of a miner's lamp, or by the blasts of the explosives used to break down the shale. Coal dust is rendered innocuous by humidity which renders it plastic and prevents its being held in suspension in the mine atmosphere. The necessary moisture is supplied either by the direct use of water, applied with a sprinkler, or by steam. In the latter case, in cold weather, the steam is used to raise the cold air entering the mine to mine temperature by means of radiators, and is then turned into the air to give it the desired humidity.

"Where it is not practicable to use steam or water, coal dust is mixed with stone or adobe dust so that there is at least 65 per cent of the latter present in the mine dust, under which conditions it will not form explosive mixtures with the air. It seems probable that the latter method will have to be used in shale mining, as indications are that shale dust does not easily combine with water.

"It will require larger quantities of explosive to break the shale than are used in coal mining, and the blasting will raise its temperature materially. It is very possible that the heat generated in blasting will be sufficient to cause a slight distillation of the lighter and more dangerous inflammable gases from the hydrocarbons in the shale. To remove such gases, as well as the smoke and gases from the blasting, will require an adequate and reliable supply of air, properly conducted to the working faces.

"With the above described conditions to be met, it will be necessary in order to secure reasonable safety, to have all blasting done by a properly qualified shot-firer after the other employees have left the mine, to use only permissible explosives, to use only electric lamps underground, and to have a mine foreman who holds a first-class certificate from the [State] Coal Mining Department."

CRUSHING OIL-SHALE.

Oil-shale must be broken or crushed before it is retorted, and the type of crusher required will naturally depend largely on the degree of fineness to which the shale is to be reduced. This in turn may depend largely on the type of retort used. As yet there is little available information as to the most suitable size of shale for retorting, and this may vary with different shales and with different retorts. (See page 183.)

For crushing, or rather breaking, toothed rolls do the work satisfactorily in Scotland, but it should be remembered that pieces of shale, often as large as a building brick, are put in a Scotch retort, and that the average size of shale retorted is fairly large. Toothed rolls should be suitable in the United States if the shale is not to be reduced to very fine pieces, but most of the proposed retorting processes intend to use shale varying in size from half-inch to 80 mesh. Reducing rich American shales to such small sizes is difficult, as the shale is tough and elastic and in the crusher tends to yield without shattering.

There is little real information available regarding the crushing of American oil-shales, but so far, the hammer-mill, ring-pulverizer, and gyratory crusher seem to be favored, with toothed rolls or ordinary jaw crushers for preliminary crushing to sizes varying from 1 to 1¼ inch. Ore-milling practice should be of assistance in solving this problem.

INTERRELATION OF RETORTING AND REFINING.

No special effort is made in this Bulletin to discuss separately the problems of retorting and refining, as it cannot be too strongly emphasized that these phases of shale-oil production are so intimately connected that one must be considered with the other.

Certain retorting conditions produce a high yield of oil, but the oil may be of relatively poor quality. Other conditions may give a smaller amount of crude oil, but its quality may be so much better than the other that it will yield a larger quantity of refined products. Certain methods of retorting will leave most of the nitrogen in the spent shale, from which it can be recovered as ammonia or ammonium compounds if desired. Other conditions will cause more nitrogen compounds to appear in the oil, from which they must be removed in refining. Certain oils of poor quality can be greatly improved by preliminary refining operations. Relatively slight changes in conditions of retorting sometimes have important influences on the nature of oil produced, and the result of a change may be of different degree with different shales.

The broad technologic problem to be considered here probably may best be stated as the determination of those conditions of retorting and methods of refining that will yield the greatest profit. Generally, this means those conditions and methods that produce at lowest cost the maximum quantity of products for which there is the greatest demand, or for which the greatest demand can be created. Thus, the problem is partly economic and its solution may be different for each individual plant.

RETORTING PROBLEMS.

Considering retorting from a broad standpoint, the first question to be considered is whether Scotch practice can be adopted entirely for American shales and conditions; must it be modified, and how; or must it be abandoned entirely. The same considerations hold with respect to refining the shale-oils. Scotch practice has never been given a fair trial on American shales, and there is much that would lead one to expect that the Scotch retort, probably somewhat modified, may be well adapted to treat many of our shales. These points have been discussed previously, so it is unnecessary to cover the ground again.

Some rich shales tend to intumesce in the retort when heated. Even Scotch shales tend to bridge and clog in the retort. The modification of the Scotch retort, or the design of new types to overcome this difficulty is a matter that must be carefully considered.

An important field for study is to determine the best type of material to be used in retort construction. This applies to refractories. When coke by-product plants were first introduced

into this country from Europe, ordinary fire-brick was used as refractory material in their construction, but American operators were not satisfied with the heat-conducting capacities of such brick, and other materials were tried. Other refractories were utilized, some of which greatly increased the rate of heat transmission from the furnace to the charge in the retort, and the new refractories resisted abraison better than ordinary fire-brick. Oil-shale operators may expect to obtain much valuable information in this regard from those who have applied such refractories to coke-oven construction, and the possibilities of using silica brick, carborundum brick, and the like in shale retorts, seem to be favorable. As for the highly heated metal parts of the retort, consideration should be given to the possibility of using recently developed alloys and protected castings, capable of standing sustained high temperatures without deterioration.

The shale-plant engineer will desire information as to various physical and chemical constants of the oil-shale which his plant is to treat. Some of these constants are specific gravity, weight per unit volume when crushed to different sizes, specific heat, heat of conductivity, heat of combustion, and proximate and ultimate analyses. These factors naturally vary in different shales, but the Bureau of Mines, co-operating with the State of Colorado, has determined them for an average Colorado shale, and they are presented in the Appendix (page 156). Knowledge of these factors is necessary, if best engineering principles are to be applied to oil-shale retorts and retorting practice. Likewise specific and latent heats of the crude oils and their fractions should be determined. Knowledge of the coefficients of expansion of the different oils will be essential.

Certain individual factors that influence retorting, and thereby the quantity and quality of products obtained, deserve careful study. Some of these factors, which are now being studied by the Bureau of Mines, co-operating with the States of Colorado and Utah[110] are—

> (1), rate of rise of temperature of shale; (2) maximum temperature to which the shale is subjected; (3), amount and temperature of steam, or other vapors and gases, such as shale gas, carbon monoxide, and the like, used in connection with retorting; (4), fineness of the shale being retorted; (5), pressure under which retorting is conducted; and (6), time of contact of vapors with retort walls.

A summary of the experimental results of the work mentioned above is presented in the Appendix (page 179).

110 Gavin, M. J., and Sharp, L. H., A study of the fundamentals of oil-shale retorting: Bureau of Mines Reports of Investigations, Serial No. 2141, June, 1920, 4 pp.

After the effects of these and other factors are determined experimentally, they must be applied to large scale work, insofar as commercial and engineering considerations permit. They will be of value in determining the particular type of retort that will provide the best conditions at lowest installation and operating costs. In commercial practice, of course, absolute optimum conditions, experimentally determined, are not always applied; but they are approached as closely as economic conditions and mechanical considerations permit.

That retorting oil-shales and refining shale-oils are intimately interrelated has already been emphasized. The production of crude oil from oil-shale is a simple operation, merely requiring the heating of the shale with exclusion of air, and subsequent condensation of vapors; but, the economic production of maximum quantities of the most valuable oils from the refiner's standpoint is not a simple, but a complex and difficult problem; therefore, the study of production of oil and other products, carried out under different conditions as outlined above, will not be complete until the products so obtained have been subjected to thorough refining and chemical study.

It is quite conceivable, from a consideration of the principles of destructive distillation, that certain conditions of retorting will produce an oil which will be practically valueless from the refiner's standpoint. Many inventors who are seeking to develop retorts with extremely large unit throughput apparently do not realize that the quality of an oil is as important, and in some cases more important, than its absolute quantity. The quantity of crude oil produced is not nearly as important as the quality and quantity of refined products that can be made from the crude. Not only must the crude oil be of good quality, but it must be of uniformly good quality, day after day. (See Appendix, page 187.)

PRODUCTS DESIRED FROM OIL-SHALES.

The success of the Scotch oil-shale industry has depended primarily on the production of paraffin wax and ammonium sulphate. Conditions relating to ammonia production in the United States are discussed on page 123, and as regards wax, the present condition of the wax market in this country is such that if any large amount were being produced from a new source, unless it were of exceptional quality, it is difficult to see where it could be marketed at profit. The demand for wax is limited, and American petroleum, even though its production gradually declines, should largely take care of that demand for many years without much difficulty. It must be realized that much of our petroleum contains considerable quantities of paraffin wax which are not now extracted, simply because the demand for wax is insufficient to warrant increased production. If, however, as is almost gen-

erally expected, American oil-shales will produce high melting-point waxes cheaply, there will undoubtedly be a demand for them.

During the past several years the petroleum products in greatest demand have been motor fuels, illuminating oils, lubricants, and fuel oils. There is also a growing demand for fuel-distillates for gas making, for certain metallurgical operations, and particularly for heavy-oil engines, especially of the semi-Diesel and Diesel types. The Diesel engine has the highest thermal efficiency of all known internal-combustion engines, and as the more volatile motor-fuels become scarcer and more costly it is natural to expect that the Diesel, semi-Diesel, and other types of motors, which utilize fuels most efficiently, will be used more and more.

The demand for illuminating oils is increasing very slowly, and no immediate appreciable increase can reasonably be expected. Lubricants will probably be supplied from petroleum for a great many years, since much of the petroleum and petroleum residuums now burned as fuel can be worked into lubricants when the demand for lubricants increases. Oil will continue to be used as fuel until its price exceeds that of an equivalent amount of coal. The demand for motor fuels is, however, likely to grow at an increasing rate. This being the case, it seems likely that, first of all, shale-oil will be used as a source of motor fuels, and secondly, possibly as a source of fuel oil. Other products will probably be of secondary importance.

American shale-oils will probably yield a relatively good percentage of volatile motor fuel, once refining methods have been developed; and apparently a large percentage of the less volatile distillates from shale-oils will make satisfactory gas-oils and heavy-oil engine fuels. It is doubtful whether satisfactory domestic kerosenes, for lamp use, can be made from shale oil, except at a cost that will be practically prohibitive for years, but probably the cheaper grades of illuminating and burning oils can be made at relatively low cost, and without great difficulty. In recent years the tendency has been to decrease the volatility of fuels for automotive engines, and the design of engines is constantly being changed so they can use the lower grade fuels satisfactorily. If this tendency continues, as it probably will with higher-priced oils, automotive fuels made from shale-oils may contain a large amount of what would now be considered an illuminating-oil distillate. This distillate probably could not be cheaply refined into a satisfactory illuminating oil, while it could be used as a motor fuel.

As is shown on page 187, the first step in the refining of shale-oil will probably be to run the crude oil down to coke. For this

reason little or no residual fuel-oil will be produced from most shale-oils. In subsequent distillations of partly refined products, however, residues may be produced which will be satisfactory fuel-oils, if they do not contain too much paraffin wax. Many of the heavy distillates which may be used as heavy-oil engine fuels or fuel-oils, may contain so much wax that it will have to be removed, or the oil heated to a temperature of complete fluidity before transportation or use.

Since motor fuels and fuel-oils will probably be the products in greatest demand, and most easily made, the greatest effort in retorting and refining should be made to obtain them, particularly the first. The market for them will undoubtedly be stable and the demand can reasonably be expected to increase steadily. Later on as the price of petroleum becomes higher, and more is known regarding the methods of refining shale-oils, it may be possible to make a more complete line of products.

As yet, it is not definitely known whether lubricating oils of high viscosity and durability can be made from American shale-oils, but investigations by the Bureau of Mines are yielding hopeful results in this regard. (See Appendix, page 160.) Although the demand for lubricating oils and lubricants generally, is, and for many years probably will be, taken care of by petroleum, the possibility of producing satisfactory lubricants from shale-oil should be given serious consideration, as sooner or later the petroleum supply may fail to such an extent that the demands for lubricants cannot be supplied by it. At present, a great deal of petroleum, which could be refined into lubricants were the demand for lubricants greater, is being burned as fuel.

Oil-shales will be expected to supply the country with many, or all, the products now derived from petroleum, when our petroleum production can no longer adequately meet the demand. Therefore the oils must yield marketable products, and the retorting conditions must be such that each shale-oil will yield maximum quantities of refined oils, consistent with economic considerations.

NITROGEN RECOVERY.

Oil-shale operations in Scotland have been successful largely on account of the high recovery of ammonium sulphate which has a ready sale as a fertilizer. Table 6 (page 30) indicates that many American shales contain nitrogen in amounts equal to or larger than that contained in the average Scotch shales, and thus the possibility of producing ammonium sulphate or other ammonia salts from domestic shales is indicated.

Little information is available as to the form in which nitrogen exists in American oil-shales, but results of investigations by the Bureau of Mines (report now being prepared), indicate that the nitrogen exists in such condition that it may be readily converted

into ammonia by the action of steam on the shales at high temperature, after the oil has been produced. It appears that those conditions found to be favorable for ammonia production by the Scotch oil-shale industry, will also be favorable for producing ammonia from American oil-shales; but conditions for producing maximum economic yields of this material from our shales are, of course, not yet definitely known, and cannot be known until commercial shale plants are in operation. It has been noted that while it is possible to convert the nitrogen of the Scotch shales into ammonia almost quantitatively, the operators in Scotland find it most economical to make only about a 60 per cent conversion.

Attention is directed to the fact that conditions of producing the oil from shale largely determine the amount of nitrogen that remains in the oil-spent shale to be acted upon by the steam at high temperature. Rapid production from the shale—rapid heating and rapid removal of vapors—yields an oil containing a relatively high percentage of nitrogen, and a spent shale with a relatively small percentage of nitrogen. Slow oil production reverses this effect, and this, evidently, is one reason why the Scotch retorts are designed to heat the shales slowly.

There is a possibility of recovering the nitrogen as nitrogen-bases, such as pyridine, pyrrol, and the like, which may be of considerable value as preservatives, germicides and insecticides, although there is little market for them at present, and because of their evil odor they must, in any event, be removed from the refined oil products before the latter are marketed. Dilute (10 per cent) sulphuric acid removes about 10 per cent of the volume of crude naphtha from Colorado shale-oil produced by dry distillation, and nitrogen-bases constitute practically all of this loss. It may be found possible to recover such nitrogen bases in a form suitable for commercial use, providing their recovery does not too greatly depreciate the value of the other products of shale-oil.

If a means of retorting and refining can be devised that will prevent in retorting the formation of oils containing nitrogen, or cheaply and easily eliminate them from the crude and its oil products, it may be well to disregard the production of ammonia altogether, since this product is being made by new and cheap methods which may render its recovery from oil-shale generally unprofitable. This may well be the case, if by neglecting ammonia production, retorting costs can be reduced, or in other words, net profits be enlarged; a larger throughput of shale per retort obtained; and the oil producing capacity of the retort be increased. A possible solution of this problem is discussed in the Appendix (page 184).

This consideration also suggests the possibility of treating the oil-spent shale in separate gas producers, independent of the re-

tort, in which it should be possible to increase ammonia recovery almost to the total theoretical amount possible, and to produce considerable quantities of gas for fuel purposes. The lower part of the Scotch type of retort is, of course, a gas and ammonia producer, but many engineers are of the opinion that gas and ammonia production may be effected more efficiently, and to better advantage, by means of a separate apparatus entirely independent of the oil-producing retort. Some suggest that by this means as much or little of the oil-spent shale as desired can be treated for the production of fuel gas and ammonia. In any event, full consideration should be given to the possible effect of any of these operations on the quality of the shale-oil produced.

EFFECT OF SULPHUR AND SULPHUR COMPOUNDS.

Oil-shales, as a rule, contain small quantities of sulphur, and it is important that the crude shale-oil contain a minimum of this element or its compounds. Sulphur compounds in the oil make refining more difficult and costly, and in some cases calls for the rejection of the crude altogether for refining or fuel purposes. It will be necessary to study the distribution of the sulphur in products made from different shales, and if sulphur compounds are present in objectionable quantities, either in the oil or gas, efforts should be made to prevent their occurrence in these products, or to insure their occurrence in forms easily removable. Practically all shales yield hydrogen sulphide when they are retorted; some produce large quantities. This gas is a dangerous poison, if present in sufficiently large concentrations, and it may sometimes be necessary to guard against its possible effects on plant workers.

DUST IN THE OIL.

Whenever oil-shale is retorted, a certain amount of dust is formed, and carried over into the condensers with the gases and vapors. The amount of dust produced varies with the shale, its condition and the method of retorting. It accumulates in the condensers and varying quantities are always found in the oil. If much is present in the oil, it must be removed before refining.

A study of retorting and the design of retorts will always take account of possible dust troubles, and provision should always be made for easy removal of dust accumulations in vapor-mains and crude-oil condensers.

REFINING PROBLEMS.

Regarding the refining of shale-oils, it has been noted that shale-oils are usually highly unsaturated; that is, they contain certain unsaturated hydrocarbons together with nitrogen bases, which must be removed in refining before the oil products can

be marketed. The amount present will depend somewhat on the method by which the oil was produced. Certain of these compounds, probably the more highly unsaturated, cause the color of the products to darken slowly and the deposition of gums and resins rendering the oils unsuitable for fuel and illuminating purposes. This behavior is noted particularly in the lighter fractions of the oil, and as stated, is probably due to polymerization and oxidation of certain of the compounds. The removal of these objectionable bodies requires several acid and alkali treatments, and redistillations in refining, and largely accounts for the high refining loss in Scotch shale-oil refineries.

In Scotland, about 25 per cent of the crude oil, including still coke, is lost in refining, as against a maximum refining loss of about 7 per cent in completely refining petroleum in the United States. Petroleum, of course, contains a much smaller quantity of unsaturated hydrocarbons than shale-oil; many petroleums contain none or only a trace of them. In spite of the long experience and economical methods used by the Scotch operators in refining shale-oils, the high refining loss can not be reduced in commercial practice. If ordinary American petroleum refining methods were used the refining loss would undoubtedly be considerably higher. It is to be noted that when there is a loss of this nature in refining oils, not only is there a loss of a certain amount of the oil, but this loss must be paid for, as it results from expensive chemical treatment, made necessary by the presence of objectionable compounds. If the crude oil originally contained less of these substances, a saving is made, both in absolute recovery of refined products and in lessened costs of treatment.

From the above discussion certain definite refining problems may be outlined:

1. Determination of the color and resin-forming compounds in the oil, and methods of removing them without removing other harmless unsaturates.

In Scotch practice not all the unsaturated compounds are removed in refining. As a general rule, the presence of olefins in motor fuels, up to a certain percentage, is other than objectionable, and therefore some of the olefins may be permitted to remain in certain of the products, while the more objectionable compounds should be eliminated.

2. Recovery of acid and alkali from sludges obtained in treating the oils.

Recovery of acid from sludges is an established practice in many American petroleum refineries, but means of recovery of the more expensive alkalis has not been perfected.

3. Improved and cheapened methods of refining.

Refining of shale-oil is at best an expensive operation. The feasibility of using continuous distilling and treating processes, and the possibility of using less expensive treating reagents, should be considered.

4. Use of sludges for purposes other than for fuel.

In Scotch practice, the sludges produced in treating the oils amount to about 16 per cent of the crude treated. After the recovery of the acid from them, the treated acid and alkali sludges are burned under the stills as fuel. In most American petroleum refineries the sludges are also burned as fuel. It would seem that there are certain possibilities in connection with the reworking of these sludges to obtain other commercial products.

5. Hydrogenation or saturation of unsaturated compounds.

If the unsaturated compounds of the oils could be cheaply saturated, there would be a smaller refining loss, and a production of more and higher grade finished products. Commercially, it has not been possible to hydrogenate mineral oils successfully, but a further study is advisable, especially in the case of oils so highly unsaturated as shale-oils.

6. The effect of coking distillations on crude shale-oils is discussed in the Appendix (page 184). It may be noted here that the effect of a coking distillation on an oil produced by rapid retorting is to yield a distillate much like the crude oil made by slow retorting. Even an oil made by slow heating can be improved by a coking distillation. A thorough study of this effect will indicate to what extent it can be used in refining, and is certain to have a decided influence on the methods used in producing the oil.

ECONOMIC CONSIDERATIONS.

Economic considerations must govern all oil-shale operations. As in the case of most industries, it is not possible to secure absolute optimum results in all the phases of oil-shale retorting and shale-oil refining. Operations will, naturally, be so conducted that the greatest net profits will be made on the whole operation of producing finished products. In each locality, with every shale and with each type of retort used, the most economic conditions for the production of the greatest profit should be determined. For example: at the present time ammonium sulphate commands a good price, while nitrogen bases have no market whatever, that is to say, no market has been developed for them; hence, under present conditions, the practice might be to make as much ammonium sulphate as possible from the shales (if nitrogen recovery is considered at all). Under reversed conditions, it would be advisable to make as much of the nitrogen bases as possible. Again, the most valuable product obtained from Scotch shale-oil is paraffin wax, but conditions of supply and de-

mand might be such that it would be advisable to leave as much as possible of the wax in the gas and fuel oils, and lubricating oils, without unduly raising their setting-points or cloud-tests. Market conditions must be studied, and the total net value of products made and marketed be such that the shale and oil treating operations, as a whole, will yield the greatest possible profit.

CONSERVATION.

Many problems, especially some of those just discussed, are not peculiar to the oil-shale industry alone. The successful solution of some of these problems by oil-shale operators can well be applied in many cases to the refining of petroleum. It may be remarked that while petroleum is cheap much of it is wasted, and refining processes were not developed with the primary purpose of utilizing a valuable resource in as efficient a way as possible; but when petroleum becomes costly, and the Nation has realized that its natural petroleum resources are not inexhaustible, more attention will be paid to conservation and prevention of waste. It would have been better to have thought of petroleum as a valuable and wasting national resource, to be utilized as efficiently as possible, when the industry began, but when a commodity is plentiful and cheap, little thought is given to the fact that in time it may not be so.

Our oil-shale deposits are large, but they are not inexhaustible. When the oil-shale industry first begins it may not be always feasible, as well as profitable, to introduce the refinements into the treatment of the shale and its products which would mean conservation of this natural resource, but the shale should not be wasted, and in considering the future the country should demand that it be efficiently utilized. To bring about this ideal condition, much research and investigative work will be necessary, to determine the products that can be obtained and the methods of producing, refining and using them. By the proper application of work of this nature, it should be possible to conserve and efficiently utilize the nation's oil-shale resources very early in the history of the industry.

BY-PRODUCTS.

Discussion of conservation and efficient utilization leads naturally to consideration of the possible by-products of oil-shale. The term "by-products" used in connection with the oil-shale industry is commonly understood to mean products other than the ordinary hydrocarbon oils and waxes similar to those now obtained from petroleum.

It might be desirable, of course, to produce by-products, if these are of sufficient commercial value. If, however, the production of by-products, for the sake of profit, is responsible for a

waste of other products, perhaps less valuable, but nevertheless necessary, then from a standpoint of national conservation such production could not be considered an economic policy. Much is heard regarding by-products, and it seems to be the general impression that the industry will succeed because of such products. All kinds of substances have been included in the list of by-products from oil-shale, such as vanillin, salicylic acid, pepsin, rubber, perfumes, medicinal preparations, drugs, and dyestuffs. For most of these "paper" derivations there is a limited demand, consequently a limited production at the present time. It should be realized that if it were possible to make these products from oil-shale on a commercial scale, the output might far exceed their consumption and demand, so notwithstanding their present high prices these might decrease to such an extent that further production would be unprofitable, if a new market for them could not be created.

In a large way, by-products are rather unstable things to serve as a foundation for an industry of any considerable magnitude, and the oil-shale industry, if it is to succeed at all as a national industry, must be one of great magnitude. Putting it more plainly: a few small individual plants might possibly operate at a profit because of the production of rare by-products, but when hundreds of large oil-shale works begin to produce it seems reasonable that the once rare by-products will become plentiful, and their market would become demoralized. This is only another view of the law of supply and demand, which must be considered in connection with the oil-shale industry, as it must be in any other. It may be true that many rare products have been produced in an experimental way from oil-shale, but there is a vast difference between the laboratory preparation of a substance and its profitable commercial production. Thus, it might be possible to produce sugar from garbage in the laboratory, but it is difficult to imagine that such a process would be a paying one, commercially.

Oil-shales can hardly be expected to produce products other than those that can be made from coal-tar and petroleum. There is now (1922) an overproduction of coal-tar in this country. More is being produced than can be worked up by the coal-tar industry, and much is being burned as fuel-oil in the Eastern States, where coal by-product practice is common. As the by-product coke industry is growing, it seems that it will have little difficulty in supplying all our requirements for products ordinarily obtained from coal-tar. Moreover, it is highly probable that if it is desired to produce oils similar to coal-tars, from oil-shales, a different method of retorting must be used than if oils similar to petroleum are desired. In other words, it seems that the conditions favorable

for the production of many substances commonly obtained from coal-tars, such as phenol, cresols, naphthalene, dye bases, and the like, will not be those favorable for the commercial production of volatile motor fuels, kerosenes, and similar products, for which there undoubtedly will be a greater demand and a more stable market. It is not certain, as yet, that oil-shales can be so treated as to yield products similar to coal-tars.

Oil-shale operators will do well to plan on making products for which there is a demand, and for which a stable market exists. Such products are likely to be those similar to the refined petroleum oils of commerce, and possibly some of the by-products of the petroleum industry for which there is a well established market. These products, and by-products, principally hydrocarbon oils similar to those produced from petroleum, are those which oil-shales are expected to supply. If it fails to do this, the oil-shale industry can not hope to become one of much importance or magnitude.

Certain exceptions are possible in regard to the above statements. Nitrogen products may, and possibly will be derived from American oil-shales. In Scotland, ammonium sulphate is not considered a by-product. Moreover it is probable that by-products may be obtained from oil-shale for which a stable demand can be created, by creating new uses, or in the case of some, for which a small demand now exists, cheaper production or the expansion of demand might make their production profitable. Notwithstanding these considerations, attention is directed to the opinion that, at this time, it seems that much of the by-product investigation and discussion is ill-timed, particularly as the industry is not fully informed as to methods of producing satisfactory crude oils, and making marketable hydrocarbon oils from them. On these latter products, the success or failure of the oil-shale industry will depend.

SCIENTIFIC RESEARCH.

In connection with the study of oil-shale, a great many investigations of a scientific nature should be made. Some of these are: the determination of the nature of the oil-producing material of the shales; the manner in which it decomposes in forming oil; the relation between mode of origin, geologic age and character of products; and the chemical nature of the oil and gas produced from different shales under different retorting conditions. Many people disparage such studies, not appreciating that results of pure research of this type often become of highest commercial importance.

ECONOMIC PROBLEMS.

The oil-shale industry, to be commercially successful, must make a return on the capital invested in it; and to attract cap-

ital, must offer reasonable evidence to the prospective investor that it will make a fair return on his investment. Some of the factors to be considered in this regard are technical, and have been discussed in other sections. Other factors are economic in nature, but, from a broad viewpoint, the technical and economic considerations cannot be wholly separated. It is not a function of the Bureau of Mines to investigate the economic side of the oil-shale industry, but a brief discussion of some of these phases may call attention to matters hitherto not always carefully considered.

LABOR SUPPLY.

The question of securing, housing, and feeding the labor necessary in oil-shale districts, when the industry begins to assume a position of some magnitude, is one to which attention must be paid. In the Eastern States the problem of securing labor may not be particularly difficult, but in the Rocky Mountain States it is a real problem, which is liable to be serious fairly early in the development of the industry.

Local labor will undoubtedly be available at the start, but as the industry grows, men will have to be hired, transported to the shale-fields, and housed in a region not now well supplied with transportation facilities, and for the most part, sparsely settled. When the industry begins to reach the magnitude of the present status of petroleum production, the number of miners alone required will be comparable with those employed in our coal mines (average of 750,000 men). The problem is not serious now, nor is it likely to be so for some time, but the ultimate probability of drawing hundreds of thousands of men from other industries, without serious industrial disturbance, and organizing an effective working force from them, is so fraught with economic problems as to deserve attention even now.

SUPPLIES.

Those who will manage oil-shale plants will have to consider the problem of securing supplies necessary in the development and operations of the industry. Iron and steel, refractories, chemicals used in refining, timber and explosives for mining, fuel and the like, must be available or made available. These may necessitate the development of industries subsidiary to the oil-shale industry, as near as possible to the shale-fields.

TRANSPORTATION.

Attention will have to be paid to the securing of transportation facilities, and to the transportation of products from the shale works and supplies to them. There are few railroads in the shale district of the Rocky Mountain States, and transportation facil-

ities as they are at present will serve for the development of only a small part of the shale resources. Much of the rich oil-shale is many miles distant from railroads. The great Uinta Basin of Colorado and Utah, for example, is now served by a single narrow-gage road, with Shay locomotives, and running over 60 miles to a transcontinental main-line connection. The grade on this line in places reaches a maximum of 9 per cent.

It is probable that most retorting plants will be constructed as near as possible to the shale mines; and where railroad facilities are, at present, not too far from the deposits, spurs and branches will probably be built to connect with the various plants. For other deposits new roads must be constructed. Those companies now operating or erecting small shale plants should consider the future growth of the industry, and plan on securing adequate transportation service for the time when the industry will become commercial.

There is a technical problem involved in one phase of the transportation problem: The chief product of an oil-shale retorting plant is crude oil, and if this oil can be transported by pipe-line from the retorting plants to central refineries on the railroads, one of the chief transportation problems will be solved. On account of the high percentage of solid paraffins in most shale-oils, as a rule, they will not flow well below temperatures ranging from 85 to 100° F. However, if heating stations are provided at intervals along the pipe-line, the line buried to proper depth and well insulated, and the oil forced through at sufficiently high velocity, it is probable that the oil can easily be pumped, even during the winter. Most shale-oils are quite fluid at temperatures only a few degrees above their setting points.

It is recommended that the possibility of piping shale-oil from groups of retorting plants at the mines to a large refinery on the railroad, be given serious consideration, not only on account of the low cost of this method of transportation of oil compared with the cost of shipping in tank cars, but because a large refinery can operate more efficiently and with smaller overhead and fixed charges per barrel than a small one. Also, such a refinery would be in an advantageous position for securing supplies and marketing products. It is believed that a 20,000-barrel refinery, situated on a railroad, and receiving oil by pipe-line from 20 retorting plants, each producing 1,000 barrels of oil daily, could operate at a much lower cost per barrel and with greater efficiency, than twenty 1,000-barrel refineries, each situated close to and serving a retorting plant producing a like amount of oil daily, and depending on spur railroad connections and tank-cars to get its products to the main rail line; or even using pipe-lines to transport refined oils to storage on the railroad.

The oil-shale districts of the Eastern States are more adequately served by rail transportation systems than those of the West, but, in all cases, due consideration will have to be given to the problem of getting marketable products to the consumers.

MARKETING OIL-SHALE PRODUCTS.

Under the best possible conditions, because of the capital and construction work required, it will be many years before an oil-shale industry begins to assume the magnitude of the present petroleum industry in this country, and therefore, many years before shale products are extensively marketed. In the next section (page 139) it will be noted that the writer does not believe that shale-oil products will ever be serious competitors of petroleum products. Shale-oil may be expected gradually to replace petroleum as the petroleum supply fails. It seems likely, therefore, that shale-oil products, when they are marketed in considerable quantity, will be marketed through the present systems, and under the conditions used in marketing petroleum.

SOME FACTORS INFLUENCING PLANT LOCATION.

Some of these factors have been discussed in the previous section, but good engineering practice will give consideration to other problems such as: position of plant with respect to mine, availability of dumping space for spent shale, water supply, layout and future expansion of plant, and housing and sanitation for employees.

POSITION OF PLANT WITH RESPECT TO MINE.

The relative positions of the plant and the mine concerns the conveyance of raw shale to the retorting plant. This will depend to a considerable extent on the topographic features of the ground on which the plant is to be constructed. In most of the Western shale regions it is probable that for a long time the shale will be mined from outcrops on cliffs, high above the valley floors, and the plants will be erected in the valley, or between mine and valley floor, to facilitate gravity flow of waste. In this case, chutes or surface trams undoubtedly will be used in conveying shale to the plant. Aerial trams may also be used. Types of all these systems are being employed in the Rocky Mountain region by the small plants now operating intermittently for experimental or promotion purposes. Surface trams or chutes will probably be most favored, as they are cheaper to install and operate than other systems. In other parts of the country it may be necessary to haul from the mine entry to the crusher bins, particularly where there is little natural slope to the ground. In such cases electric locomotives or cable systems may be used to haul the shale in cars from the mine directly to the crusher bins. Later

on, shale may be mined by shafts from the tops of the mesas, and the plants may also be situated there. (See page 113.)

DUMPING SPACE FOR SPENT SHALE.

Scotch oil-shale expands in volume during the retorting process, but most of the richer American shales contract somewhat while being retorted. Therefore, nearly, if not quite, as great a volume of waste material will have to be disposed of as is put into the retorts. Some use may be found for the spent shale, but with large scale operations it is not likely that a market can be established for spent-shale products sufficient to take care of any great proportion of this waste material.

Assuming that a ton of crushed raw shale occupies 35 cubic feet, and the volume does not change in retorting, a plant of 1,000 tons daily capacity will discharge about 476,000 cubic yards of spent shale in a year, equal to a cone about 580 feet in diameter and 145 feet high. (See Plate XIII for size of dumps at one of the Scotch works.) Provision must be made for disposal of this large quantity of waste at the lowest possible cost. In many districts the plant can be so placed as to discharge directly onto a steep hillside, but in others it will be necessary to provide conveying or haulage systems to move the spent shale to dumps. (See Plate XIV.) This is an item which must be considered in selecting plant sites and calculating operating costs.

WEATHERING OF SPENT SHALE.

Many American spent shales, when exposed to the weather, rapidly disintegrate and settle down, probably because of their high content of calcium and magnesium oxides, and turn into a black sticky mud, which would probably be washed away and later be deposited as silt during rainstorms. Attention of shale operators is directed to this point, as this characteristic of spent shales may call for special attention in their disposal. The writer believes that it may be necessary later on for the States, in which oil-shale deposits occur, to enact laws to enforce the disposal of spent shales in such a manner that properties at a lower elevation may not be damaged. Mr. J. J. Jakowsky, to whom the writer is indebted for data on experiments on the weathering of spent shales, is convinced that the weathering of such shales may present serious problems to oil-shale operators.

WATER SUPPLY.

In many cases the problem of securing adequate and reliable supplies of water for oil-shale retorts and refineries will be serious. This difficulty may not often be encountered in the Eastern States, but the shale fields of the West are in districts where rainfall is usually sparse, and water-courses few and rather

widely scattered. In some places it is probable that water can be developed by sinking wells. Some operators plan to bring water from the larger streams to the shale works, but frequently this will require extensive piping and pumping systems. In many districts a large part of the stream flow has already been appropriated for agricultural purposes, and further irrigation projects are planned or can reasonably be anticipated, in potential farming areas near the shale fields or below them on the same river systems.

Retorting and refining operations will probably require from 150 to 250 gallons of water per ton of shale treated. Water may be used as steam in the retorts; probably water-cooled condensers will be used, especially in regions where seasonal temperatures range from 100° F. in summer to below zero in winter; boiler plants require water; refinery condensers must certainly be water-cooled; water may be required in mines; and a good supply of potable water will be needed for domestic purposes by the large number of employees and their families. It seems certain, in many cases, that water supplies will have to be carefully conserved; exhaust steam will be condensed; condenser water will be cooled in towers or ponds and re-used; and heat exchangers will be employed partly to take the burden of cooling from the condenser water. It will probably be advisable in most cases to use electric power transmitted from hydro-electric plants on the larger rivers to the shale works and mines, at least in the Western States. Possibly excess combustible gases from the retorts, or producer gas made from raw or spent shale, can be used in internal-combustion engines to generate power at the plants. There is also a chance that at least part of the water required in the works can be obtained from the shales themselves. Many shales when heated will yield 30 gallons and more of water to the ton, and if this water is sufficiently pure it may be used for boilers and condensers.[111]

PLANT LAYOUT AND FUTURE EXPANSION OF PLANT.

A shale retorting-plant and oil-refinery should be arranged for economy of space and operation. Provision should be made for expansion, and the frequent error of petroleum refinery engineers in failing to allow for plant enlargement should be avoided from the start. Naturally, the first shale plants will be comparatively small, but the assumption is that they will grow, and extensions should not be haphazard. A convenient, logical, and economic flow-sheet should be designed at the very beginning, and allowances made so that enlargements of the plant will permit con-

111 J. J. Jakowsky, Assistant Oil-Shale Engineer of the Bureau of Mines, has prepared a comprehensive paper on "Uses of water in the oil-shale industry", which will be published in the near future.

formation with the same flow-sheet. Many engineers do not realize the extent production costs are lessened by convenient internal arrangements of a plant, which afford logical and orderly flow of raw materials to the works, flow of products in the course of manufacture from stage to stage, and output of finished products and waste materials from the plant. Work of this type calls for a high degree of engineering skill. An industry that can hope for only a relatively small margin of profit cannot pay too much attention to this point.

This matter makes necessary the consideration of position of retorting plant with respect to mine, railroad, refining plant, and storage facilities. Frequently the ideal condition of having mine above retorts, retorts above refinery, and refinery above storage, can be realized. This will allow gravity flow of raw, finished, and waste material, and construction can be planned so that subsequent enlargements will not disturb the efficiently planned flow-sheet.

HOUSING AND SANITATION.

It is not out of place to discuss the problems of housing for employees and camp sanitation.

Industry, as a whole, is rapidly appreciating that a contented worker means an efficient worker. Nothing assists more in bringing contentment than comfortable and clean living quarters, and sanitary and wholesome living conditions. The point has already been made that labor will have to be brought into many of the shale fields from remote points, and into a sparsely settled district. Conditions must be made attractive to the workers, if it is hoped to retain their services for any extended period. Living quarters should, therefore, be made comfortable, clean and attractive; well-constructed and inviting homes should be available for the married workers; wholesome and substantial food should be provided, and mess houses should be well lighted and clean. Opportunities should be afforded for wholesome recreation and amusement.

Camp sanitation should receive careful attention. Water supplies may often be scanty and, particularly in these cases, proper sanitation will require utmost watchfulness. Disposal of waste and sewage must also be carefully considered, not only for the benefit of the individual camp, but for the protection of others at a lower elevation along the same stream or lower down the same valley.[112]

---

112 The subject of sanitation in oil-shale camps is fully discussed by Murray, Arthur L., Sanitation in planning and developing oil-shale camps: Bureau of Mines Reports of Investigations, Serial No. 2265, July, 1921, 7 pp.

THE FUTURE OF THE OIL-SHALE INDUSTRY IN THE UNITED STATES.

The future of the oil-shale industry in this country depends primarily upon the relative supply of, and demand for, petroleum products, particularly in the regions remote from seaboard. As already indicated, there is good reason to believe that in the next several years the domestic production of petroleum will decrease, while the demand for its products will increase, and oil-shales can well make up for the deficiency of crude petroleum as a source of refined mineral oil products.

No one expects the oil-shale industry to spring into being overnight, as a full-grown industry of national importance and comparable with the present petroleum industry. Even under the most favorable conditions its development must be slow, although this can be hastened by the employment of highly-trained specialists, the proper kind of experimental work, and sincere co-operation and mutual helpfulness among the oil-shale operators.

There are certain practical limitations to the rapid development of the oil-shale industry that should be mentioned. M. L. Requa, former Director of the Oil Division of the United States Fuel Administration, has stated[113] that the oil-shale industry developed to the scale of the present petroleum industry "would require a mining activity comparable in size to the coal mining industry". George Otis Smith, Director of the United States Geological Survey, speaking of the possibilities of the oil-shale industry, makes the point that "plainly our country can not afford to support another such army of workers (as that of the coal-mining industry), until we reach another stage in our industrial development".[114]

Large sums of money will have to be invested before the oil-shale industry becomes one of important commercial consideration, altogether aside from the problem of securing capital. It is probable that the investment necessary for an oil-shale retorting and refining plant will approximate $3,000 per barrel of shale-oil daily capacity. The present annual domestic output of petroleum is at the rate of over 400,000,000 barrels, and to replace that production with shale-oil would require nearly 1,100 shale retorting plants, each putting through 1,000 tons of shale

113 Requa, M. L., The petroleum problem: Bulletin of the Union Petroleum Co., Philadelphia, 1920, p. 26.
114 Smith, G. O., Where the world gets its oil: Nat. Geog. Mag., Vol. 37, Feb., 1920, p. 197.

daily, every day in the year. This is assuming that the shale yields 42 gallons of oil per ton. The total quantity of shale mined would be over 400,000,000 tons, which approaches the annual coal production. The investment for retorts and refineries alone for an industry of this magnitude would be over $3,000,000,000. This does not include estimated cost of lands, opening up and developing mines, or transportation and marketing facilities; neither does it include the cost of developing subsidiary industries, without which a shale-oil industry could not exist.

COMPARISON OF THE OIL-SHALE WITH THE PETROLEUM INDUSTRY.

It is probable that the investment represented by our present petroleum industry is of somewhat the same degree of magnitude as the estimate given in the preceding section as the requirement for an oil-shale industry which would replace the present domestic petroleum production with shale-oil. The petroleum industry, however, offers a more attractive form of investment than does the oil-shale industry at the present time, if both investment charges and costs of producing oil in each case are compared. Profits are often large and rapidly acquired in the petroleum industry, while only a conservative profit can be expected from oil-shale operations, at least for a great number of years. Large interests will not invest heavily until convinced that oil-shale operations will be profitable, and attractively profitable. On the other hand, once a shale-oil industry becomes profitable, profits are assured, subject to nothing more than ordinary business risks. There will be little of the spectacular in the oil-shale business, but ultimately it will be a conservative industry, in which to invest for conservative, though probably sure, returns. Statements of this nature have been disputed by many of the present-day oil-shale enthusiasts, but the writer has seen no reliable estimates or figures that would cause him to alter his views.

It must be understood, however, that the industry will undoubtedly grow slowly, and while doing so, it will become more successful. One plant will begin operating at a fair profit, after which capital can be more readily attracted. However, one plant, favorably situated, with favorable markets, and properly financed and managed, may begin to make money in a relatively narrow marketing field, while others, not so well favored, might have to wait years for profits.

The oil-shale industry is not strictly comparable with the petroleum industry. It is still an embryonic industry in this country, and calls for much new training and experience. Obtaining oil from shale is not comparable to obtaining petroleum

by drilling. In drilling an oil-well the element of chance has always to be taken into consideration, but there is always the possibility of large immediate reward on a relatively small investment. There should be no such element of chance in producing shale-oil under proper management. In the first place, a few well-placed drill-holes on the land selected will make possible the estimation of the thickness and quality of the deposits. After that, the production of crude shale-oil becomes a technical problem, that of producing a crude product from a low-grade ore. The production of oil from shale requires one more operation than does the production of petroleum. In the latter case, once the mine (well) is driven into the oil-sand, the crude product is recovered with but relatively little cost. In the case of oil-shale, the rock itself must be mined and afterwards treated to produce the crude oil. The character of petroleum is what it happens to be when the driller strikes the sand; the character of shale-oil is largely dependent upon the process and conditions used in its manufacture. Once the crude shale-oil has been produced, its refining is more complex and more costly than the equivalent refining of petroleum.

The oil-shale industry will ultimately be developed on as safe and sane a basis as other great manufacturing and mining industries. It is clear, however, from experience in other countries, and from the very nature of the mineral and its crude products, that the industry can come to commercial importance only as a type of large scale, low-grade raw materials mining and manufacturing industry, and the profits derived from it will be of the order of those derived from other industries of the same general nature.

It is hardly believed that shale-oil, considered in a large way, will be a competitor of petroleum; it is more likely to be a slowly developed successor of petroleum. A general impression has arisen that the larger oil companies are seeking to retard development of the oil-shale industry, because they fear the possible competition of shale-oil. There can be little truth in this belief. The writer believes that the petroleum industry as a whole welcomes the advent of shale-oil, as its leaders are fully aware of the serious situation the American petroleum industry is facing. The larger, and many of the smaller companies, are already considering the time when they will be producing shale-oil or handling its products. Many have already acquired oil-shale lands. Competition of shale-oil with petroleum can not be regarded seriously, since, because of the many technical problems which must be solved in connection with the former and the large amount of capital which will be required before the industry can hope to compare in production with the present petroleum industry, shale-oil will probably be sorely needed long before it

is produced in quantities sufficient appreciably to relieve the impending shortage of petroleum. It will be much longer, as before noted, before it can go far in supplying the present demand for petroleum, which normally will grow at a much greater rate than domestic petroleum supplies can support. M. L. Requa, former Director of the Oil Division of the United States Fuel Administration, at the November, 1920, meeting of the American Petroleum Institute, in an address, made a statement[115] which is worth quoting in this regard: "The oil-shale industry, the coal-refining industry, the power-alcohol industry, with their potentialities and their limitations, deserve our close consideration. While they may superficially appear as our competitors, they are fundamentally our allies. When the time is ripe, I believe these supplemental sources of supply can be developed by the petroleum industry more advantageously than by any other agency".

Shale-oil appears to be the most natural and logical substitute for petroleum. The supplies of shale are so great as to dwarf by comparison the quantity of petroleum already produced and still available for production in the United States. The writer believes that the oil-shale industry will ultimately be an industry of great magnitude and commercial importance in this country, but many years and much money will be required before it reaches this status. In its last analysis, it is an industry comparable with the low-grade-ore mining industries of the Western States, and like them, will require the services of the highest types of business, executive, and technical skill, backed by large capital, and which can afford, and be prepared, to wait a considerable time for a conservative return on the investment.

The development of the oil-shale industry should be promoted legitimately and honestly. It can be best developed, and the right kind of backing encouraged by presenting facts and not half-baked theories; by deliberately recognizing and facing the limitations and problems of the industry, instead of glossing these over with a coating of vague estimates and statements of the enormous possibilities of the industry. It can be aided also, by the proper kind of investigation, and making public the results of investigations by governmental and private agencies; and by the proper kind of co-operation of governmental agencies, petroleum organizations, and shale-oil companies. The embryonic oil-shale industry should, for the sake of rapid growth, and its future, clear its skirts of the swarm of fraudulent promoters and promotion companies which is so large at present that the true position and merits of the oil-shale industry are difficult for many to see.

115 Requa, M. L., Conservation: Am. Pet. Inst. Bull. No. 132, December 10, 1920, p. 58.

ESTIMATES OF COSTS AND PROFITS IN THE OIL-SHALE INDUSTRY.

It is unnecessary to caution those who have had experience in estimating or calculating costs and profits in any large-scale manufacturing industry in regard to making similar estimates for the oil-shale industry. However, much promotion literature has appeared, giving estimated costs and profits of this or that oil-shale company operating, or contemplating operations, and similar estimates have appeared from time to time in the technical and non-technical press. In all but very few cases these estimates have neglected to include many important items of cost, which must be considered if an estimate of even fair accuracy is to be obtained.

In the first place, the present status of our oil-shale industry is such that it is impossible to make reliable estimates of costs. Practically all retorting and refining installations, intended to treat oil-shale and shale-oil, are so small that they indicate little as to their operation under commercial conditions, or if they will work at all under such conditions. No one really knows just what products can, or will, be made from oil-shale, what price these products will bring in a competitive market, or with what degree of satisfaction they can be used. Rates of depreciation of equipment can not be determined accurately with small and non-commercial plants for application to large plants working under commercial conditions. There are other items entering into the profit and loss statement which can not be determined with even a rough degree of accuracy at the present time. It must be realized that economic and competitive conditions will be different in different districts, and that it is, therefore, impossible to make a blanket estimate to cover operations in any or all districts, as has been frequently attempted.

For these reasons the writer does not venture to estimate probable costs and profits for the future American oil-shale industry. Some factors influencing these items will be the type of process used; nature and extent of shale deposits; completeness of refining the crude products; scale of operations; situation of plant with respect to supplies, water, and markets; local cost of labor; and transportation costs and facilities. Such factors may be very different for different companies, and must be considered for each plant.

It may be worth while to point out some items entering into cost estimates for the oil-shale industry, many of which have been

frequently overlooked or ignored by those who have publicly issued statements of estimates.

SOME ITEMS TO BE CONSIDERED IN ESTIMATING COSTS.[116]

Capital Investment.—The main items of capital investment will consist of the following:

(a) Cost of land, in which may be included legal expense in connection with clearing of title; cost of surveying; water-rights; permanent improvements, such as road building; and cost of sampling and preliminary assays.

(b) Buildings, including those necessary for mining, crushing, storage and retorting of the shale, and for refining the products. This item will also include machine-shop, laboratories, garage, office, buildings for housing labor and the like.

(c) Machinery and equipment, including that necessary in mining, retorting and refining operations; power plant equipment; laboratory equipment; garage equipment (machinery and automotive equipment); machine-shop equipment; furniture and fixtures for office, laboratory, and camp; and transportation equipment (tanks, barrels, drums, tank-cars, pipe-lines, trucks, and the like).

Manufacturing Expense.—Under this heading are grouped many items of expense frequently overlooked in making estimates. Manufacturing expense includes the following:

(a) Material. Cost of raw material is either the purchase price of the land or the rentals and royalties on leased land. If the land is purchased it constitutes a wasting asset, and depreciates in value for every ton of shale mined. Under this heading may also be included costs of supplies, chemicals, fuel, water, etc., used in mining, retorting, and refining, and cost of their delivery to the plant. Sulphuric acid and caustic soda will probably be used in refining, and these chemicals constitute an important item of cost.[117] There may also be included under this heading the cost of packing and preparing products for market, although this may better be charged to sales expense, or plant overhead.

(b) Direct Labor. Labor entering into the direct production of shale-oil and its products. It should be distributed properly for each stage of mining and manufacture.

(c) Overhead Expense. This includes indirect labor; superintendence; depreciation of plant and equipment; heat, light and

---

116 The material in this section is based on a report, "Some items of investment, expense, and profit in oil-shale operations"; Bureau of Mines Reports of Investigations, Serial No. 2214, Feb., 1921, prepared by L. H. Sharp and A. T. Strunk, of the State of Colorado Co-operative Oil-Shale Laboratory, Boulder, Colorado, with the assistance of the writer.

117 Losses in refining shale-oil will be greater than losses incurred in the equivalent refining of petroleum. Such losses must be charged to the particular stage of manufacture in which the loss occurs, and the amount of such loss based on the value of the refined, not the crude, product.

power; taxes; insurance; repairs and maintenance; interest on value of products in storage; miscellaneous supplies; laboratory maintenance and miscellaneous expense. Overhead should also be distributed properly for each stage of mining and manufacture.

Selling and General Expense.—The main items of selling and general expense would be somewhat as follows: Salaries of general office force and sales force; advertising; commissions; insurance on office buildings, fixtures, and sales equipment; office supplies and maintenance; taxes on office building and sales equipment; heat, light, and power for office building; depreciation of office building, furniture, fixtures, and distributing and sales equipment; repairs on, and maintenance of, delivery and sales equipment; interest and expense on unpaid obligations; traveling expenses; cost of legal services and accident insurance or compensation for employees. Shrinkage in quantity of products between refining and selling point should be charged to selling expense.

The above does not pretend to be a complete statement of items of investment and expense for the industry, but it may serve to indicate that many of the hitherto published cost estimates for oil-shale operations are not as comprehensive as they should be.

SOURCES OF PROFIT.

The probable sources of profit in the oil-shale industry are crude oil or its products, which ultimately are expected to be motor fuels, burning oils, gas and fuel oils, lubricants, paraffin wax, nitrogen compounds derived from oil, shale gas (which may be an indirect source of profit by reducing fuel costs, as also may refinery sludges if burned under stills), and possibly ammonium sulphate or similar ammonia salts. There may also be important by-products and specialties, but, as yet, it is not safe to calculate on them.

The total income from these sources must be sufficient to cover manufacturing, marketing, and general expense, and leave a margin for depletion reserves and for profit on capital invested. Income must be calculated on the basis of current refinery prices, not retail prices. The difference between these two, less transportation and sales expenses, will be the profit of the retail sales division, if the organization has its own retail sales agency, and if there is any profit to be made in retailing.

APPENDIX

PART I—SAMPLING AND ASSAYING OIL-SHALES.

SAMPLING.

Before a deposit of oil-shale is opened up, the operator will desire to determine the probable quantity of oil and other products obtainable from the shale, also to decide on what part of the deposit to work. After operations have started, it will be advisable to make assays on samples taken from the mine, and from new workings, to serve as a check on commercial retorting operations, and to ensure that the shale sent to the retort does not go below a certain minimum richness.

Probably the most important factor entering into the value of an oil-shale assay is the method used in taking and preparing the sample. The accuracy and reliability of any analysis depends primarily on the fairness of the sample taken; that is, whether it accurately represents the deposit sampled. Samples must be so taken as to accurately represent, in proper proportions, the entire deposit under consideration. It is not particularly easy or simple to obtain a representative sample from any shale deposit.

In sampling shale it is important to take the samples from beyond the zone of surface weathering, and for this reason it is recommended that they be taken by core-drilling. However, especially in the case of massive deposits, surface weathering apparently does not affect the shale for more than a few inches from the outcrop, and in many cases, therefore, it should be satisfactory to cut a trench, perpendicular to the bedding plane of the seam, and across the entire face of the seam. This trench should be of uniform width and depth, and material taken from the first 6 or 8 inches from the outcrop should not be included in the sample. In this case it is recommended that a sample of at least 100 pounds be taken, crushed, and quartered down to 25 pounds. This quantity should be crushed to maximum size of ½ inch and again quartered to 5 pounds, which should be crushed to maximum size of ¼ inch, thoroughly mixed, and used for assaying.

Sampling at a working face could be done in the same manner, though it would not be necessary to reject the surface material, and in many cases a head sample of 25 pounds should be sufficient.

For sampling a working face, it is recommended that a trench 4 by 4 inches be cut, clean and uniform, perpendicular to the bedding-plane of the seam. In sampling a trench care should be taken not to break off very large pieces of shale, as the richer material has a tendency to break in larger pieces than that of lower grade.

Generally the methods commonly used[118] for mine sampling, as described in any standard textbook on mining, should be adhered to. The accuracy of the assay depends on the accuracy of the sample.

ASSAYING OIL-SHALES FOR OIL YIELD.

Many methods have been proposed for determining the oil-yield of samples of oil-shale for use in the field and in the laboratory. In a previous publication[119] of the Bureau of Mines on oil-shale, the assay method used in Scotland was described, and tentatively recommended to American oil-shale investigators. However, the bureau has for some time been studying methods for testing and assaying oil-shales, and has developed a method and apparatus which is more convenient to use than that developed in Scotland, and is of apparent greater accuracy, especially when working on the richer American shales. The yields obtained by the new apparatus from Scotch shales are about 2 gallons higher per ton than those obtained by the Scotch method on the same shale, but the quality of oil produced is about the same with each method. The new method was devised by L. C. Karrick, of the Bureau of Mines, after trying out practically all of the proposed types of assay apparatus. For the purpose of this Bulletin, it will be sufficient to quote from a report[120] describing the apparatus and method of using it.

---

118 The following publications of the Bureau of Mines describe recommended methods of sampling coal:

Pope, G. S., Sampling coal deliveries and types of Government specifications for the purchase of coal: Bull. 63, 1913, 63 pp.

Pope, G. S., Methods of sampling delivered coal, and specifications for the purchase of coal for the Government: Bull. 116, 1916, 64 pp.

Holmes, J. A., The sampling of coal at the mine: Tech. Paper 1, 1911, 18 pp.

Fieldner, A. C., Notes on the sampling and analysis of coal: Tech. Paper 76, 1914, 59 pp.

Pope, G. S., Directions for sampling coal for shipment or delivery; Tech. Paper 133, 1917, 15 pp.

119 Gavin, M. J., Hill, H. H., and Perdew, W. E., Notes on the oil-shale industry with particular reference to the Rocky Mountain District: Bureau of Mines Reports of Investigations, Serial No. 2256, April, 1921, p. 19.

120 Karrick, L. C., A convenient and reliable retort for assaying oil-shales for oil yield: Bureau of Mines Reports of Investigations, Serial No. 2229, March, 1921, Revised April, 1922, 6 pp.

Figure 4 illustrates the assay retort now used by the bureau. The parts necessary to set up the apparatus are indicated, and may be purchased from any good chemical or assay supply-house. By observing the following directions results of duplicate determinations can be expected to agree within 1 to 1½ per cent in the field, and within less than 1 per cent if the apparatus is used in the laboratory:

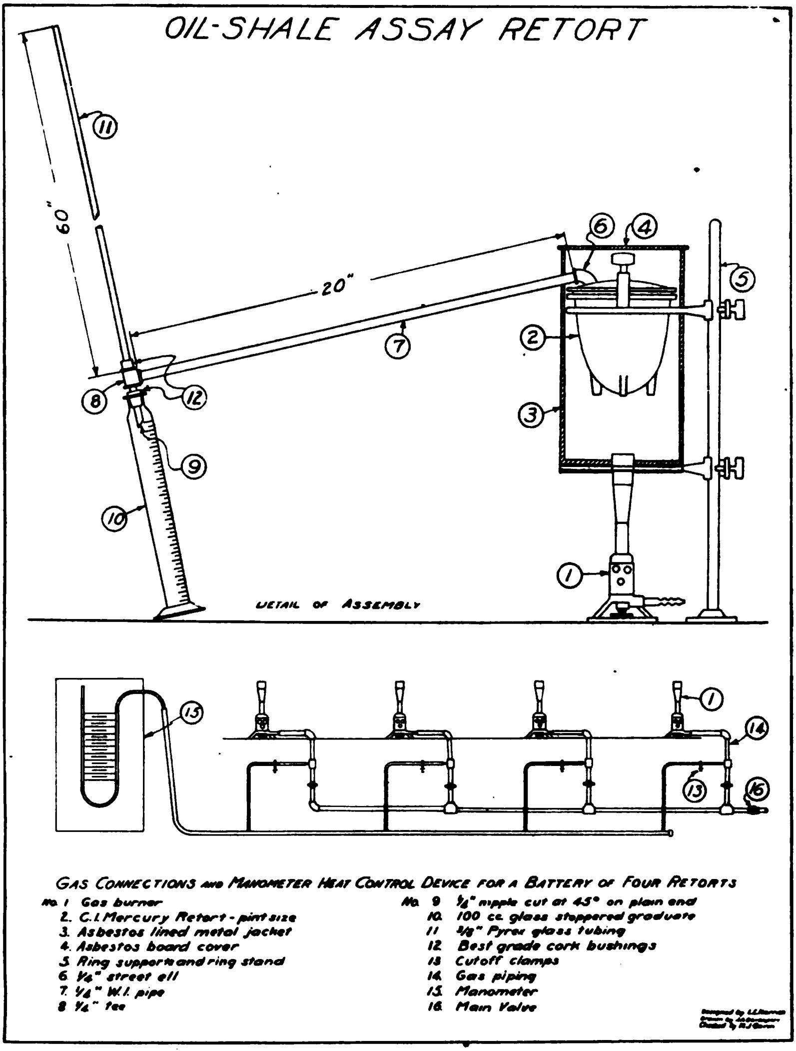


Figure 4

METHOD OF OPERATING ASSAY RETORT.

"**Sealing of Retort Important:** In assembling the outfit, all pipe threads should be turned up tightly with a mixture of pulverized litharge and glycerin. The connections (12) with the reflux condenser (11) and with the graduate (10) should also be well sealed with the same cement unless the highest grade corks are used.

"Extreme care should be exercised in the preparation and maintenance of the contact surfaces of the lid and base of the retort. These surfaces will not form a gas-tight fit when new, and this defect must be remedied by grinding the two parts together by hand, using No. 220 carborundum and water, as in grinding cylinder valves. The grinding is facilitated by clamping the lid in an inverted position on a table and pressing down lightly on the base while rotating it. After grinding, clean the parts thoroughly with clean water, clamp the lid in place, then test the improved contact by immersing the retort in water, and forcing air into the delivery tube, with the lungs. If small air bubbles appear, the leakage must be repaired. Try readjusting the lid, and if this does not stop the leakage, then repeat the grinding. The first preparation of the contact surfaces may require one-half to an hour's time, but once an air-tight contact is produced, a slight grinding before each run will keep the surfaces in perfect condition. The improved surfaces will form a gas-tight contact if kept perfectly clean and aligned while tightening the clamp.

"It is unnecessary to use a cement between the contact surfaces when they are properly prepared, and it is recommended that no cement be used. All the cements tested for this purpose have been found to be unsuitable, either because they fail to seal the joint, or adhere so tenaciously that difficulty is experienced in cleaning the surfaces for subsequent runs, or become more or less porous on heating, causing loss of oil by capillarity. A clean, well prepared emery-ground joint is convenient and reliable, and when tested as above described, assurance can be had that no oil has been lost during the run through leakage of vapors. After a little experience has been gained, each retort can be ground and made ready for a run in five minutes.

"It is important to use a jacket around the retort while heating, permitting about one-quarter of an inch clearance at the sides and high enough to clear the clamp screw on top. Holes must be cut in the jacket for ring support and delivery tube. A cover of asbestos board is necessary to reflect heat down against the retort lid, otherwise much oil will condense on the inner side of the lid, and drip down onto the shale in the retort, thus causing losses by secondary distillation.

"**Operation of the Retort.** Crush the shale so that all will pass a one-quarter inch screen or smaller mesh. Mix the samples thoroughly and then weigh out just enough to fill the retort (2) level full. Tamping the charge into the retort is permissible, and will not affect the results; this, however, is seldom necessary, and experience has demonstrated that it is better to permit the charge to rest loosely in the retort, as some rich shales intumesce or coke together, and removal of the residue in such cases is usually difficult. After the lid is fastened carefully in place and tested for leakage as directed above, place the retort in ring support (5), adjust jacket (3) and cover (4), start the burner (1), then fasten the graduated cylinder (10) and reflux condenser (11) tightly in place. Use a very small flame at first, so that no oil will appear within one hour after starting. One hour is about the minimum time in which the shale charge can be brought up to the initial distillation temperature and yet possess a fairly even temperature throughout. Some shales begin to yield vapors at approximately 250° C., whereas others show no signs of oil vapors until 375° C. is reached. Before distillation makes much progress, the interior or coolest shale should be at least as hot as the vaporizing temperature of the oil evolved, otherwise vapors will migrate to the central or coolest part and condense there, causing losses of oil by cracking on redistillation of the condensed oil.

"When about one cubic centimeter of oil has accumulated, the flame should be increased. If the maximum quantity of oil is desired, it is imperative to distill rapidly, as soon as oil appears in the receiver. If the flame has been of such size that oil first appears in about 60 minutes, it will then be safe to double the size of the flame, and thereafter every 20 to 40 minutes to increase the heat by an equal amount. The oil should accumulate in the graduate at a uniform rate, and the analyst will soon become adept at pushing the rate of retorting without exceeding the condenser capacity. It should be remembered that the quantity of oil recovered will vary for the same shale sample if the rate of retorting varies, the greater quantity being obtained with rapid retorting, and conversely, a much less amount with a long distillation period. With the average oil shale, the recovery when the actual oil-producing period is approximately 2 hours, will be 5 per cent greater than when a 6-hour distillation period is used. When distillation is completed in less than 2 hours, there is danger of inaccuracy from exceeding the condenser capacity, and from decomposition of oil vapors by excessive temperatures. When heat is being supplied very rapidly, the temperature surrounding the distilling shale particles may be much higher than is required for further oil production. This condition is brought about by the low thermal conductivity of the shale, and by the heat consuming reactions that take place as destructive distillation progresses inwardly from the surface of each shale particle toward its center. The surface of the shale particle will, therefore, be the hottest part, and it is through this hot zone that the vapors must pass in finely separated capillary streams in escaping from the porous spent shale. This condition will cause some cracking and loss of oil. Distillation should be rapid, however, when the maximum yield of oil is desired, in order to prevent stagnation of oil vapors within the hot retort or connection, and in all cases should progress at a uniform rate. If oil droplets appear near the top of the reflux condenser (11), the rate of heating must be retarded slightly. The condenser has been made small in order to limit to a safe maximum the rate of retorting that will assure the greatest recovery of oil of high quality from the average oil-shale.

"If distillation has progressed uniformly, the rate of oil accumulation will decline very suddenly as the last oil is being distilled off, and, when this cessation becomes apparent, the flame should once more be increased the usual amount, which will be sufficient to finish the distillation. White vapors may appear at the end of the run, but these have been found to be non-inflammable and to carry no oil vapors. The delivery-tube should be kept warm throughout the run in order to prevent the oil from congealing therein and to save delay in draining at the end of the run. When it is observed that the rate of accumulation of oil has suddenly declined and the bottom of the retort has become a very dull red, it can be safely assumed that the distillation of oil is completed, as this temperature is well above the final oil-yielding temperature for all oil-shales. The heating should continue for a half hour longer to permit complete drainage of the delivery-tube and reflux-condenser.

MEASUREMENT OF THE OIL RECOVERED.

"**Volumetric Method.** Inexperience in measuring the accumulated oil will cause a much greater error in results than will a lack of refinement in retorting technique, or imperfections in the retort.

"The stoppered graduate and contents must be warmed until the oil is in a very fluid condition. Allow the oil to settle well; some water is given off near the last stages of distillation, and will remain suspended in the partially congealed oil. In order to read accurately the volume of the oil and water, it is necessary to assist the formation of a perfect

meniscus by releasing any oil and water clinging to the sides of the graduate. This is best accomplished by revolving the graduate, while still warm, between the palms of the hands. Read the upper level of the oil and then the lower level, if it is well defined and is not rendered obscure by emulsion, sediment, or clinging oil and water. The following procedure will facilitate accurate reading of the lower meniscus, and also provide against the possibility that water and sediment may not have settled out completely. With a pipette draw off all but a few cubic centimeters of the oil while still warm; dilute the contents of the graduate with ten to twenty cubic centimeters of clean gasoline, and agitate gently till the emulsion disappears. Allow the solution of shale-oil and gasoline to settle, then draw off and discard it; add gasoline as before. Repeat as often as required, until a clear meniscus results. From the number of cubic centimeters of oil collected, calculate the gallons of oil per ton of shale.

"**Gravimetric Method.** To determine more accurately the amount of oil recovered, weigh the graduate and contents and allow sufficient time to settle well, while warm. (The weight should be expressed in grams.) Remove the oil and determine its weight by the method described below. Draw off with a large pipette all the oil except 4 or 5 cubic centimeters, being extremely careful that no water or sediment is drawn into the pipette. Set this sample aside for specific gravity and other determinations as it will be a sufficiently representative sample of the total oil recovered. Dilute the remaining few cubic centimeters of oil with 20 cubic centimeters of petroleum ether, and while adding the ether, wash down the inner sides of the graduate with the solvent. When all the water and solids have settled and the shale-oil is well mixed with the petroleum ether, draw off the clear liquid, nearly to the level of the water and discard it. Repeat this washing process several times until a colorless solution of ether remains on top of the water and no shale-oil is left clinging to the walls of the graduate. Remove the ether to approximately the level of the water and eliminate the balance by evaporation. The last of the ether is very quickly removed by tipping the graduate as far as possible to one side without spilling the contents, then revolving the graduate while blowing into it; the ether is picked up on the side-walls of the graduate during rotation, thus providing a large evaporating surface. As soon as the ether disappears, which will require scarcely a minute's time, weigh the graduate, now containing the water and sediment only, and subtract this weight from the original weight; the difference will be the total weight of the oil obtained from the shale. Determine the specific gravity of the oil first removed, with the Barrett, Regnault, or similar type of specific gravity bottle, and divide the total weight of oil obtained by the specific gravity. The result will be the true volume of oil in cubic centimeters, at the temperature of the specific gravity determination. (Ordinarily 60° F.) This is converted to gallons of oil per ton of shale by use of the formula:

$$\text{Gallons of oil per ton of shale} = \frac{\text{Cubic centimeters of oil} \times 240}{\text{Grams of shale used in retort}}$$

"Duplicate volumetric determinations should agree within two per cent and gravimetric determinations within one per cent.

"The table of factors given in Table 25 below, will facilitate calculations. For any given weight of shale used (column 1 or 2), select the corresponding factor in column 3. Divide by this factor the volume of oil collected (in cubic centimeters) in order to convert into gallons of oil per ton of shale. For shale charges whose weights in grams are not even multiples of ten, it will be necessary to interpolate to obtain the proper factor"

TABLE 25—Factors facilitating calculations of oil-shale assays.

Weight of Retort Charge				Weight of Retort Charge		
Grams	Ounces	Factor		Grams	Ounces	Factor
1	2	3	Column No.	1	2	3
10	.35	.042		310	10.94	1.294
20	.71	.083		320	11.30	1.335
30	1.06	.125		330	11.65	1.377
40	1.41	.167		340	12.00	1.419
50	1.76	.209		350	12.36	1.460
60	2.12	.250		360	12.71	1.502
70	2.47	.292		370	13.06	1.544
80	2.82	.334		380	13.41	1.586
90	3.18	.376		390	13.77	1.627
100	3.53	.417		400	14.12	1.669
110	3.88	.459		410	14.47	1.711
120	4.24	.501		420	14.83	1.753
130	4.59	.542		430	15.18	1.794
140	4.94	.584		440	15.53	1.836
150	5.29	.626		450	15.88	1.878
160	5.65	.668		460	16.24	1.919
170	6.00	.709		470	16.59	1.961
180	6.35	.751		480	16.94	2.003
190	6.71	.793		490	17.30	2.045
200	7.06	.835		500	17.65	2.086
210	7.41	.876		510	18.00	2.128
220	7.77	.918		520	18.36	2.170
230	8.12	.960		530	18.71	2.212
240	8.47	1.001		540	19.06	2.255
250	8.82	1.043		550	19.41	2.295
260	9.18	1.085		560	19.77	2.337
270	9.53	1.127		570	20.12	2.379
280	9.88	1.168		580	20.47	2.420
290	10.24	1.201		590	20.83	2.462
300	10.59	1.252		600	21.18	2.504

Plate XVIII is an alignment chart prepared to facilitate calculations of results with the assay retorts. Larger-scale copies of this chart can be obtained from the Director of the Bureau of Mines, Washington, D. C.

Figure 4 also illustrates a method of connecting up a battery of four assay-retorts to the same gas supply, so as to permit simultaneous distillations with all retorts. The manometer-system of connections provides a means of observing instantly the gas pressure at each burner and therefore each burner can be regulated to give any desired heat. This system is in use in the oil-shale laboratories of the Bureau of Mines and provides accurate heat control when operating the four retorts simultaneously, either at one rate or separate rates.

If an oil or activated-charcoal scrubber is used with the assay retort, scrubber naphtha can be recovered from the average shale at the rate of 1 gallon or more per ton. This naptha is a valuable product, and should be taken into account if accurate results are desired. If an oil scrubber is used, it is connected to the upper end of the reflux condenser by a short piece of rubber tubing, and the gas is drawn through the oil by an aspirator or water suction-pump connected to the outlet of the scrubber bottle. A manometer must be placed in the line between condenser and scrubber to make certain that a slight negative pressure is maintained. If the gas produced with the oil is being collected in a

gasometer, the discharge of the gas-scrubbing bottle can be connected to the gasometer, and the counter-weight of the latter adjusted to create a slight negative pressure in the system. A pinch-clamp on the rubber tubing between scrubber and gasometer permits close regulation of pressure in the retort condenser system. All connections must, of course, be tight when this arrangement is used.

Any good type of gas washing bottle can be used. About 200 cubic centimeters of scrubber ("mineral-seal") oil should be used. Usually more accurate results will be obtained if two retorts are operated simultaneously, passing the gas from both through the scrubber. After distillation is completed the scrubber oil is placed in a 300 cubic centimeter still of the Engler type and the naphtha slowly distilled off. The condenser should be cooled with a mixture of ice and water, and the distillate collected in a cooled, graduated receiver. When almost all the naphtha has been distilled from the oil, a ring of oil will be seen, gradually creeping up the neck of the distilling flask. When this ring has come within an inch of the side outlet, or delivery tube, distillation should be stopped. The yield of naphtha should be calculated according to the formula:

$$\frac{\text{Gallons of naphtha}}{\text{per ton of shale}} = \frac{\text{Cubic centimeters of naphtha recovered} \times 240}{\text{Grams of shale used in retort}}$$

If two retorts have been connected to one scrubber, the total weight of shale in both should, of course, be used in the formula.

Fresh scrubber oil should be used in each determination, and it should have an initial boiling point not lower than 450° F.

In the laboratories of the Bureau of Mines activated charcoal is used as an absorbing medium. This can be obtained by placing orders with any chemical supply house, although few carry it in stock. A glass or metal cylinder of about 100 cubic centimeters capacity, and having inlet and discharge connections at opposite ends, is filled with activated charcoal specially prepared for gas scrubbing. Plugs of cotton are placed at each end of the cylinder to keep the charcoal from passing into the inlet and discharge tubes. All connections should be tight. The absorber is connected to the end of the reflux condenser of the assay retort with a short piece of rubber tubing. (If desired two retorts can be connected to one absorber.) With this kind of absorber it is not necessary to use a pump or aspirator to draw the gas through.

After the run has been completed, the absorber is disconnected, and superheated steam at about 570° F. is passed into one end, while the other is connected to a metal condenser cooled with ice-water. The steam should be passed through slowly, and at the end the whole body of charcoal should reach the temperature

mentioned above. The steam and naphtha will condense together, and separate cleanly in the graduated receiving cylinder, which should be immersed in a beaker filled with shaved ice and water. After accumulation of naphtha ceases, steam should be passed through for ten minutes more, when the charcoal will be in condition for re-use. Yield of naphtha is calculated by means of the formula given above.

ESTIMATION OF AMMONIA YIELDS FROM OIL-SHALE.

It is a relatively simple matter to determine the total nitrogen percentage of an oil-shale, and from this to calculate the theoretically maximum yield of ammonium products—usually ammonium sulphate. It is not definitely known, as yet, if it will be desirable to attempt to recover nitrogen—as ammonia or other products—from American oil-shales, but there is a fair probability that nitrogen will be recovered in some form. Lacking knowledge of the processes that will be used in domestic oil-shale operations, and how adaptable these processes will be for making nitrogen products, or what particular nitrogen products will be made, in the opinion of the writer it would seem most advisable at the present time, to do nothing more when analyzing oil-shales than to determine the total nitrogen.

Although it is highly improbable that in commercial practice all the nitrogen in an oil-shale can be converted into marketable products, yet knowledge of the total nitrogen will give a good indication of the value of shales as sources of nitrogen products, whatever they may be. Until more work has been done on the problem of recovering nitrogen from oil-shales, the writer does not believe it advisable to recommend any analytical or testing method for oil-shales that will give results expressed in terms of commercial yields of any particular nitrogen product, for the reason stated above. If total nitrogen is determined, it is possible to calculate it into terms of ammonium sulphate (or any other expected product), and perhaps for the time being, take 60 per cent of the theoretically maximum recovery, as representing possible commercial production. In Scotland 60 per cent of the nitrogen of the shale is recovered as ammonium sulphate.

The common methods used for determining total nitrogen in coals, oil-shales, and the like, are usually modifications of the Kjeldahl method, well known to organic chemists. The particular modification of this method, used with good results by the writer, is the Kjeldahl-Gunning method, and as applied to oil-shales, is as follows:

METHOD FOR DETERMINATION OF TOTAL NITROGEN.[121]

Samples are carefully quartered, and one-quarter is ground to pass a 60-mesh sieve. One gram of this fine material is digested with 30 cubic centimeters of concentrated sulphuric acid, 5 grams of potassium sulphate, and 0.5 grams of mercury, in a 500 cubic-centimeter Kjeldahl flash, for 4 hours, or until the shale is perfectly white. It is advisable to place a small funnel in the neck of the digestion flask to prevent spattering of the liquid during the digestion. After the flask has cooled for 10 minutes, add a few crystals of potassium permanganate, to ensure complete oxidation.

After thorough cooling, the solution is diluted to 200 cubic centimeters with distilled water, and 25 cubic centimeters of potassium sulphide solution (40 grams potassium sulphide per liter) is then added to precipitate the mercury. One gram of powdered zinc and a small piece of paraffin are next added to prevent bumping and foaming during the distillation operation. Then enough saturated sodium hydroxide solution to make the contents of the flask distinctly alkaline, is added cautiously. Danger of loss of ammonia can be minimized, when adding the alkali, by holding the flask in an inclined position, and allowing the sodium hydroxide solution to form a layer below the lighter acid solution. The mixture is not shaken until the flask is connected to the condenser.

The flask is now connected to a regular Kjeldahl condenser, with a condenser tube of block tin and with a Kjeldahl connecting-tube between flask and condenser. The delivery tube of the condenser is connected by a piece of rubber tubing to a short length of glass tubing, which extends below the surface of an acid solution in a 500 cubic-centimeter Erlenmeyer flask. The acid solution consists of 10 cubic centimeters of 0.25 normal sulphuric-acid solution, diluted to 40 cubic centimeters with distilled water.

The glass delivery tube is barely allowed to touch the surface of the acid in the Erlenmeyer flask, until after the distillation flask has been shaken, to prevent the acid being drawn back into the condenser and distillation flask immediately after shaking. After shaking the distillation flask to mix its contents thoroughly, the gas burner beneath is lighted, and as soon as the contents begin to boil, the glass delivery-tube is lowered to its full extent in the Erlenmeyer flask and should then dip from ¼ to ½ inch below the surface of the acid solution therein.

The rate of distillation is so regulated that 200 cubic centimeters are distilled into the Erlenmeyer flask in about 2 hours;

---

121 This method is essentially that used by the Bureau of Mines for determining nitrogen in coal, as described in Technical Paper 8, Methods of analyzing coal and coke, by Stanton, Frederic M., and Fieldner, Arno C. 1913, pp. 24-25.

then the Kjeldahl flask is disconnected, and the glass delivery tube thoroughly washed into the Erlenmeyer flask with distilled water. The excess acid in the Erlenmeyer flask is then titrated back with standard 0.1 normal ammonium hydroxide solution, using methyl orange as an indicator. The strength of the standard ammonia is carefully checked against the acid solution each day.

Blanks are run at frequent intervals during the course of analyses, using 1 gram of sugar, in place of the shale, using the same proportions of all other reagents; time of digestion; and the like, as when an analysis is made; and the correction, as indicated by these blanks, is made on all subsequent shale analyses. It is particularly necessary to run blanks after a new solution of any of the reagents used is made up. Boiling the sodium hydroxide solution for three or four hours, after the solution has been made up, and before it has been used in the analysis, will usually make the blank correction very small.

Results can be calculated to percentage of nitrogen, or directly to pounds of ammonium sulphate per ton of shale. Ordinarily it is convenient to calculate to percentage of nitrogen (N_2). Percentage of nitrogen, multiplied by the factor 94.3, gives pounds of ammonium sulphate per ton of shale. On the basis of this method of analysis, or any method that determines total nitrogen, such calculated yield of ammonium sulphate must, of course, be considered as theoretical maximum recovery, probably never attainable in commercial practice. To calculate percentage of nitrogen to commercial yields of ammonium sulphate, based on Scotch working practice, multiply percentage of nitrogen, as above determined, by 94.3 and then by 0.60.

DIRECT METHODS OF DETERMINING AMMONIUM SULPHATE YIELDS IN COMMERCIAL PRACTICE.

In an earlier publication[122] of the Bureau of Mines on oil-shale, the method used in Scotland for determining probable commercial yields of ammonium sulphate from oil-shale was described. The bureau, in co-operation with the Department of Metallurgical Research of the University of Utah, has for the past two years been studying this, and other methods, of testing oil-shales for ammonia recovery. With the apparatus under discussion, by changing conditions only a little, it has been possible to obtain very different results with the same shale. For this method to be of much value, operating conditions must be definitely fixed and rigidly adhered to, and these conditions will probably differ for different shales.

122 Gavin, M. J., Hill, H. H., and Perdew, W. E. Notes on the oil-shale industry, with particular reference to the Rocky Mountain District: Bureau of Mines Reports of Investigations, Serial No. 2256, April, 1921, p. 21.

As an example of the necessity of fixing definite directions for operating the Scotch tube-method for determining ammonium sulphate yield, the directions given for operating the tube include the statement, "* * * the iron tube is brought to a bright red heat as rapidly as possible." The diversity of opinion among operators as to what shade of color represents a bright red is sufficient to make a difference of over 10 per cent in the yield of ammonia from a given shale by this method. However, the Scotch tube-method and its modifications are proving of value in determining conditions most favorable for nitrogen recovery from oil-shales, and the Bureau of Mines is therefore continuing their use. With the tube properly equipped with pyrometer control it may be useful for assay purposes later on, when more is known concerning the commercial methods that will be used for treating American oil-shales.

The completed work on various methods of testing oil-shales for ammonia recovery, and on conditions favorable for maximum ammonia recovery, will be the subject of a Technical Paper of the Bureau of Mines.

PART II—PHYSICAL AND CHEMICAL DATA OF COLORADO OIL-SHALE.[123]

The data presented below are for a sample of massive oil-shale of the Green River formation, taken near De Beque, Colorado, and are probably representative of the average shale of this formation. Certain factors, of course, will not hold well for Green River shales that are much richer or leaner than this particular sample, and it is not probable that these data will apply to oil-shales from different districts or geologic horizons.

1. Weight per unit volume:

Size of particles	Weight per cubic foot, pounds
Uncrushed (run of mine)	53.775
Plus 1 inch	54.775
Minus 1 inch	56.015
Minus ¼ inch	58.200

2. Apparent specific gravity:
   2.00 to 2.10

3. Specific heat:

Raw shale	0.265
Shale spent by dry distillation	0.223

4. Heat of combustion:

	Calories per gram
Raw shale	2460
Shale spent by dry distillation	600

5. Thermal conductivity:

Mean, 25 to 75° C.	0.00382

6. Proximate analysis of raw shale:

	Per cent.
Loss at 110° C. (moisture)	0.60
Loss on ignition	40.00
Ash	59.40
	100.00

7. Analysis of ash:

	Per cent.
Silica (SiO_2)	44.70
Iron and alumina (Fe_2O_3 and Al_2O_3)	25.60
Lime (CaO)	17.65
Magnesia (MgO)	5.28
Undetermined	6.77
	100.00

8. Oil yield: 42.7 gallons per ton. Specific gravity of oil: 0.905.
9. Heat of combustion of oil from this shale: 10,215 calories per gram (18,387 B. t.u. per pound).

123 Gavin, M. J., and Sharp, L. H., Some physical and chemical data on Colorado oil-shale: Bureau of Mines Reports of Investigations, Serial No. 2152, August, 1920, 8 pp.

PART III—ANALYTICAL DISTILLATION OF SHALE-OILS.

Experimental study of oil-shale retorting involves the production of oil from different shales under different controlled sets of conditions, and determination of the quantity and quality of recovered products. For this work the Bureau of Mines uses several types of retorts and with each systematically alters retorting conditions in the direction indicating best results as regards oil production, considering both quantity and quality of the crude oil. The problem, briefly, is to determine those conditions which from any shale will produce the highest yield of the best oil. In the investigations, shales typical of the deposits in different states are being tested.

At the completion of each experiment the crude products are examined. This involves analyses of gas, water, spent shale, scrubber products and crude oil. Naturally, of these products, the crude oil is most important.

It is ordinarily a simple matter to determine the amount of oil yielded by a shale, but the determination of its relative quality is a more complex matter. Thus far the most satisfactory method found for determining the quality of the shale oils is an analytical fractional distillation of the crude oil and examination of the fractions of such distillation.

The method of distillation used by the Bureau of Mines, is with minor modifications, that used by it for examining petroleum[124], and is briefly described below:

NATURE OF THE LABORATORY DATA.

"The methods employed in testing and analyzing the samples are to be described in detail in a bulletin that is now in process of preparation. For the present, discussion is confined to a brief statement of the nature of the methods, and to the significance of the figures obtained. The following data are recorded for each sample:

"1. The specific gravity at 60° F. and the corresponding Baume gravity (based on the modulus 140). Actual measurements were in most cases made by the use of a specific gravity balance.

"2. The percentage of sulphur. This was determined by burning a weighed quantity of oil in a calorimeter bomb, precipitating the sulphate formed as barium sulphate, filtering, igniting and weighing.

"3. The percentage of water. If any water was present it was determined by the distillation method.

"4. Distillation at atmospheric pressure with data for the percentages distilling within 25° C. (45° F.) limits up to 275° C. (527° F.), and the specific and Baume gravities of the fractions. Distillations were made by the Bureau of Mines Hempel distillation method, using charges of 300 c. c. of oil and distilling through a 6.5 inch fractionating column.

---

124 Dean, E. W., Properties of typical crude oils from the Eastern producing fields of the United States: Bureau of Mines, Reports of Investigations. Serial No. 2202, Jan., 1921, p. 2.

"5. Vacuum distillation at a reduced pressure of 40 mm. 'absolute', of the residuum from the "air distillation", with data for the percentage distilling between 25° C. (45° F.) limits up to 300° C. (572° F.) and for the specific and Baume gravities. Saybolt universal viscosities (at 100° F.) and Fahrenheit cloud tests of the distillation fractions.

"6. Conradson carbon residue percentages of the residuum from the combined 'air' and 'vacuum' distillations."

ADDITIONAL TESTS ON SHALE OILS.

Up to the time data for this Bulletin were assembled, the Bureau of Mines had not always determined percentages of sulphur in the shale-oils. Therefore the analyses in Table 26 (page 164) do not always include percentages of sulphur in the oils. At present, however, all crude shale-oils are analyzed for sulphur content. It has been found that nearly all shale-oils contain rather large percentages of sulphur, the average of those thus far examined being about 0.6 per cent. There is, however, considerable variation from this figure, some oils running somewhat lower and a number much higher.

Also, until recently, Conradson carbon residue tests have not always been made on shale-oil residuums: consequently these are not given in all the analyses in Table 26.

On account of the high percentage of solid paraffin wax in many crude shale-oils and shale-oil fractions, it has been found somewhat inconvenient always to determine viscosities at 100° or 130° F. For this reason some of the viscosities reported were determined at 140° F. (60° C). Viscosities of the crude oils are also reported.

Specific gravities of the fractions solid or semi-solid at 15.56° C. (60° F.) are determined with a Regnault specific gravity bottle.

The dark color of many of the vacuum fractions, and of the crude oils themselves makes the taking of the cloud test impossible. Consequently on those fractions, and on the crude, a "setting point" test is made. This method also is of value in that it follows the method used in Scotland, and serves there as an index of the amount of solid paraffin wax recoverable from the oil. The procedure is to freeze a drop of the oil, of rather definite size, on the tip of the cooled bulb of a thermometer, then to invert the thermometer, and while rotating it about a vertical axis, to allow the temperature to rise at a definite rate, until the drop melts and flows down the bulb. Unless the oil is of a very tarry or asphaltic nature, the setting point can be sharply and accurately determined. Recently the Bureau has developed a somewhat more accurate setting-point testing apparatus. This consists of a horizontally placed glass tube through which water flows under constant head. A resistance wire passes through the tube and by regulating the volume of water and current flowing in the resistance any temperature gradient desired can be set up

between the ends of the tube. With the tube cool, a line of the oil to be tested is drawn on the top of the tube, the current is turned on and adjusted until the temperature of one end of the tube is above that of the setting point of the oil. A blast of air at low pressure is then directed on the line of oil, which blows away if the oil is fluid. A point is reached where the oil adheres to the tube, and knowing the distance of this point from one end of the tube and the temperature of both ends, the temperature at this point may be easily calculated. A setting-point thus determined agrees closely with one determined with a thermometer as described above.

In addition to these tests, shale-oil fractions resulting from the distillation at atmospheric pressure are combined, and an unsaturation test is made in the combined fractions. The method used and the value of the data thus obtained are discussed under the heading "Evaluation of Shale-Oils" (p. 160).

INTERPRETATION OF DISTILLATION DATA.[125]

"The methods employed by the Bureau of Mines for the distillation analysis of crude petroleum have not been developed with the idea of obtaining figures that parallel the results of actual refinery practice. As refinery practice has never been standardized, it has been necessary to select a fundamentally reproducible basis of comparison, rather than attempt to work in terms of yields and properties of commercial products.

"The chief value of the present report lies in the fact that it permits a reasonably adequate comparison of different crude oils on the basis of fundamental physical and chemical properties. The suggested method of use is most satisfactorily indicated by a typical example which is given in a later connection.

"It is believed while the most satisfactory use of the figures involves a comparison, there is also a need for some sort of 'rough and ready' interpretation in terms of commercial products. Therefore, the author has employed the following classification which, even if it has no other justification, is convenient because it permits discussion in terms of 'given names'.

"1. The sum of all fractions distilling at atmospheric pressure below 200° C. (392° F.) is reported as **gasoline and naphtha.**

"2. The sum of all fractions distilling at atmospheric pressure between 200° C. (392° F.) and 275° C. (527° F.) is reported as **kerosene.**

"3. The sum of all vacuum distillation fractions having Saybolt viscosities (at 100° F.) of less than 50 seconds is reported as **gas oil.**

"4. The sum of all vacuum distillation fractions having Saybolt viscosities (at 100° F.) between the inclusive limits of 50 and and 99 seconds is reported as **light lubricating distillate.**

"5. The sum of all vacuum distillation fractions having Saybolt viscosities (at 100° F.) between 100 and 199 seconds inclusive is reported as **medium lubricating distillate.**

"6. The sum of all vacuum distillation fractions having Saybolt viscosities (at 100° F.) of 200 seconds or more, is reported as **viscous lubricating distillate.**

"7. The suitability of a given type of crude for producing cylinder stock has been assumed to be approximately in inverse proportion to the percentage of carbon obtained by the Conradson carbon residue test made on the residuum from the vacuum distillation. This carbon residue test

125 Dean, E. W., Properties of typical crude oils from the Eastern producing fields of the United States: Bureau of Mines, Reports of Investigations, Serial No. 2202, Jan., 1921, p. 3.

undoubtedly does not tell the whole story but its general value is indicated by the fact that Pennsylvania and West Virginia crudes show Carbon residue of residuum figures between 1 per cent and 3 per cent while the corresponding figures for Kentucky crudes are from 5 to 8 per cent and for California crude from 13 to 20 per cent. Pennsylvania and West Virginia crudes are, of course, regarded as the most suitable material for producing cylinder stock, whereas it is made from oil of the type of Kentucky crude only through the use of special processes, and from average California crudes not at all."

Caution should be used in interpreting vacuum distillation data of shale-oils. Many of the vacuum fractions appear to have properties satisfactory for the production of lubricating oils. Little is known, however, as to how these fractions would stand refining, or how satisfactory for commercial use the refined lubricating oils would be. The high carbon residues of many shale oils indicate that the production of satisfactory lubricants from them might present a most difficult problem.

In any event, shale-oil lubricants are much different from petroleum lubricants. Many shale-oils yield fractions with lubricating properties much superior to those indicated by their viscosities, but the whole question of lubricating oil production from shale-oils is yet in an indeterminate state.

THE EVALUATION OF SHALE-OILS.

Attempts to evaluate shale-oils, even for comparative purposes in the laboratory, involve both technical and economic considerations. From a technical standpoint one must consider and determine what possible products can reasonably be expected from shale-oils. From the economic side there must be considered which of these possible products will be in greatest demand, and how cheaply they can be placed on the market. Possibly in some sections of the country certain products will be in greater demand than in others, and a discussion of how cheaply these products can be produced and marketed leads to a consideration of plant location, transportation, labor supply, and similar economic factors which have been discussed in a previous section, but which will present problems that each plant must individually solve.

Inasmuch as the chief problem in the oil-shale work of the Bureau of Mines is to determine the conditions most favorable for the production from different shales of highest yields of best products, it must first consider what these products are likely to be; second, determine the relative quantities of these products in the oils under consideration; and finally, determine the relative amounts of these products from different shales. The question has been given careful thought, and the matter taken up with many producers and refiners of petroleum in the United States.

During the past several years the petroleum products in greatest demand have been motor fuels, illuminating oils, lubricants, and fuel oils. The demand for illuminating oils is increasing very slowly, and no immediate appreciable increase can reasonably be expected. Lubricants will probably be supplied from petroleum for a great many years, since much of the petroleum and petroleum residuums now burned as fuel can be worked into lubricants when the demand for lubricants increases. Oil will continue to be used as fuel until its price exceeds that of an equivalent amount of coal. The demand for motor-fuel is, however, likely to grow at an increasing rate. This being the case, it seems likely that, first of all, shale-oil will be used as a source of motor fuel, and secondly, possibly as a source of fuel-oil, Other products will probably be of secondary importance, and it has been finally decided that shale-oils may reasonably be evaluated on the bases of the amount and quality of motor fuel they will produce.

An analytical distillation can indicate the relative amount of motor fuel of a given volatility to be obtained from different oils, but it is necessary to be somewhat arbitrary in selecting the range of volatility of the motor fuel, or in other words, its "end point." In making analytical distillations of petroleum and shale-oil, it is the practice of the Bureau of Mines to stop the "air" or atmospheric pressure distillation when the vapor temperature in the neck of the fractionating column reaches 275° C. The combined fractions distilling up to this temperature are considered, for purposes of comparison, as representing all the gasoline, crude naphtha and crude kerosene. The general tendency during the past few years has been to increase the boiling range, or in other words, decrease the volatility of motor fuels. This tendency has been checked for the present, but if there should be a marked increase in the demand for motor fuels, it might be renewed in the future.

No definite prediction, therefore, can be made as to the future end point of motor fuel; accordingly it has been found most satisfactory for purposes of comparison to classify shale-oils on the basis of relative percentages distilling up to 275° C. vapor temperature. This distillate is generally spoken of as "tops". Thus the value of a shale-oil as a producer of motor fuel may tentatively be indicated by the percentage of tops that can be produced from it. It is to be noted that this is taken to indicate the **quantity** of **crude** motor fuel to be expected from a given shale-oil.

Another point to which attention must be directed is the suitability for refining of this crude motor fuel fraction. Probably

this can most conveniently be studied by determining the percentage of "unsaturation"[126] of this fraction.

For the purpose of this discussion the term "unsaturates" or "unsaturated hydrocarbons" is taken to mean that part of the oil soluble in concentrated sulphuric acid under the conditions of the test above referred to. If used with this understanding, unsaturates probably includes the diolefins, and part, but not necessarily all, of the olefins. Some of the branched chain hydrocarbons may possibly be sulphonated also, under the conditions of the test. The acid also removes basic compounds, and many shale naphthas contain up to 10 per cent of nitrogen bases. Likewise by "saturated hydrocarbons" or "saturates" is meant that part of the oil not soluble in sulphuric acid under the conditions of testing.

Just what part percentage of unsaturation will play in the refining of shale-oil is not well known at present. Certainly under commercial practice the refining loss of any distillate will be much less than the absolute unsaturation percentage. However, it is probable that the relative refining losses will be approximately proportional to the respective percentages of unsaturated hydrocarbons in the oils under consideration. For example, a shale-oil containing 50 per cent of unsaturated hydrocarbons will, on refining, suffer a greater loss than will an oil containing only 25 per cent of unsaturates, but the loss probably will not be twice as great.

There is much to be learned regarding refining losses of shale-oils and the relationship between refining loss and unsaturation, but for the present and for purposes of comparison it seems most feasible to rate qualities of shale-oils largely on the basis of their percentages of unsaturated hydrocarbons.

It is a very difficult problem to determine accurately, by the usual test with sulphuric acid, the percentage of unsaturated hydrocarbons in a crude shale-oil. The acid tars produced under this treatment are usually very viscous and difficult to separate from the oil. On the other hand, it is relatively simple to determine this unsaturation percentage in the tops, and to secure on this product closely agreeing results on successive unsaturation tests. Therefore, and again tentatively, the Bureau of Mines compares the **qualities** of shale oils on the basis of **relative percentages of unsaturates in the tops from the oils.**

Another sound reason for classifying oils on this basis is brought out in the analyses. A low percentage of unsaturated hydrocarbons in the tops generally indicates more desirable

126 For a description of the method used to determine percentage of unsaturation, see Bureau of Mines Tech. Paper 298, Methods for testing petroleum products, 1922, p. 55. When shale-oil distillates are thus tested the bottle of acid and oil must be kept cool under the tap or in a pail of water while being shaken.

properties in the remaining fractions of the oil. In some cases the reduction of unsaturates seems to decrease the percentage of solid paraffin wax in the oil, but even in this case the crystalline structure of the wax apparently is altered in a direction that would make its recovery more feasible.

Knowing the amount of tops yielded by the crude oil, and the quality of the tops as indicated by its percentage of unsaturates (also its percentage of saturated hydrocarbons, since this is obtained by subtracting percentage of unsaturates from 100), an index number tentatively evaluating the shale-oil may be readily calculated. If **percentage of tops** from a shale-oil be multiplied by the percentage of **saturated hydrocarbons** in the tops, the product is a good numerical index of the value of that oil. If the possible oil yield of the **shale** has been determined by a standard method, then the crude-oil yield of the shale, calculated to gallons per ton, multiplied by this index number, gives another number which can be used as an index of the **value of the shale**, disregarding, of course, certain economic considerations referred to above.

It should be repeated that this system of classification does not take into consideration the value of the oil other than as a producer of motor fuel, although, as noted, a high yield of motor fuel fraction coupled with low percentage of unsaturates in this fraction, is reflected in the quality of the other fractions from the oil. Likewise, it does not take into consideration the absolute volatility range of the motor fuel fraction, or the percentage of sulphur in the oil. These matters require further study, and perhaps the method will require revision when these factors are properly taken into account. Nevertheless, the method has been found extremely useful to the bureau's chemists in their shale investigations, especially in studying a particular shale.

An application of the method described above may be given. Consider two shales, one yielding 50 and the other 35 gallons of crude oil to the ton. The oil from the first yields 38 per cent of tops containing 55 per cent of unsaturates (45 per cent saturated), and the other 35 per cent of tops containing 40 per cent of unsaturates (60 per cent saturated). First, evaluating the oils:

Relative value of oil from shale No. 1 = 38x0.45 = 17.10.
Relative value of oil from shale No. 2 = 35x0.60 = 21.00.

If the shales themselves are to be evaluated, the numbers obtained as above are multiplied by the respective crude-oil yields of the two shales:

Relative value of shale No. 1 = 17.10x50 = 855.
Relative value of shale No. 2 = 21.00x35 = 735.

Investigations now under way may develop a method for evaluating the heavier fractions of shale-oils. The Conradson carbon residue percentages of the vacuum residuums may possibly be used for this purpose, once the relationship between this and the quality of the heavier fractions has been determined.

Distillation analyses of typical shale-oils are presented in Table 26, following. Sample 101 is oil made from Scotch shale by Bureau of Mines assay retorts and Sample 102 is oil produced by commercial retorts in Scotland. The analysis of a typical Pennsylvania crude oil (sample 1107) is included for purposes of comparison between typical shale-oils and a typical high-grade petroleum. The other analyses are discussed and some conclusions drawn from them in Part IV of the Appendix, which follows Table 26.

TABLE 26—Distillation analysis of shale-oil.

Sample No. 305a.

Shale from Grand Valley, Colo.
Specific gravity oil, 0.891.
Viscosity at 130° F., 45.
Per cent tops, 53.2.
Per cent water, trace.

Retort: Horizontal rotary; first fraction of oil.[a]
Baume gravity, 27.1.
Setting point, 18° C. (65° F.).
Unsaturation tops, per cent, 40.7.
Index No. oil, 31.55.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 626 mm. First drop, 47° C. (117° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 130° F.	Setting point °F.
Up to 50	Up to 122	tr.					
50—75	122—167	1.2	1.2	0.728	62.3	...	...
75—100	167—212	1.9	3.1				
100—125	212—257	4.0	7.1	.758	54.7	...	...
125—150	257—302	6.1	13.2	.782	49.0	...	...
150—175	302—347	7.9	21.1	.810	42.8	...	...
175—200	347—392	6.3	27.4	.834	37.9	...	...
200—225	392—437	8.1	35.5	.855	33.7	...	...
225—250	437—482	8.5	44.0	.874	30.2	...	...
250—275	482—527	9.2	53.2	.884	28.4	...	...
Vacuum distilation at 40 mm.							
Up to 200	Up to 392	3.6	3.6	0.891	27.1	39	below 32
200—225	392—437	6.1	9.7	.907	24.4	46	39
225—250	437—482	6.0	15.7	.922	21.8	57	60
250—275	482—527	4.7	20.4	.940	18.9	82	78
275—300	527—572	7.6	28.0	.958	16.1	241	94

Residuum: Specific gravity 1.002; Baume gravity 0.2.[b]
Carbon residue, per cent, 9.4.
Residuum asphaltic.

---

[a] This is the first fraction of oil from the retort taken during a run in which a total of 3572 cc. of crude oil was produced. This fraction consisted of 1033 cc., or 28.9 per cent of the total oil produced.

[b] Baume heavy scale.

TABLE 26—Distillation analysis of shale-oil (continued).

Sample No. 305b.

Shale from Grand Valley, Colo.
Specific gravity oil, 0.904.
Viscosity at 130° F., 45.
Per cent tops, 41.9.
Per cent water, trace.

Retort: Horizontal rotary; second fraction of oil.*a*
Baume gravity, 24.9.
Setting point, 22° C. (72° F.).
Index No. oil, 22.58.
Unsaturation tops, per cent, 46.1.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer 628 mm. First drop 48° C. (118° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 130° F.	Setting point °F.
Up to 50	Up to 122	tr.					
50—75	122—167	1.1	1.1	0.716	65.5	...	...
75—100	167—212	1.7	2.8				
100—125	212—257	3.5	6.3	.760	54.2	...	...
125—150	257—302	4.7	11.0	.788	47.7	...	...
150—175	302—347	4.8	15.8	.811	42.6	...	...
175—200	347—392	5.3	21.1	.836	37.5	...	...
200—225	392—437	6.0	27.1	.858	33.2	...	...
225—250	437—482	6.6	33.7	.879	29.3	...	...
250—275	482—527	8.2	41.9	.898	25.9	...	...
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	2.4	2.4	0.909	24.0	40	below 32
200—225	392—437	7.1	9.5	.916	22.8	48	41
225—250	437—482	8.3	17.8	.928	20.9	65	60
250—275	482—527	11.0	28.8	.941	18.8	107	76
275—300	527—572	13.2	42.0	.953	16.9	252	91

Residuum: Specific gravity 0.993; Baume gravity 11.0.
Carbon residue, per cent, 8.1.
Residuum asphaltic.

a Second fraction from retort, 1384 cc. or 38.7 per cent of total oil produced in run. See footnote to sample 305a.

TABLE 26—Distillation analysis of shale-oil (continued).

Sample No. 305c.

Shale from Grand Valley, Colo.
Specific gravity oil, 0.9025.
Viscosity at 130° F., 40.
Per cent tops, 48.0.
Per cent water, trace.

Retort: Horizontal rotary; third fraction of oil.*a*
Baume gravity, 25.1.
Setting point, 22° C. (71.6° F.).
Unsaturation tops, per cent, 45.4.
Index No. oil, 26.21.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 629 mm. First drop, 40° C. (104° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 130° F.	Setting point °F.
Up to 50	Up to 122	0.7	0.7	0.708	67.7	...	...
50— 75	122—167	2.2	2.9			...	...
75—100	167—212	2.2	5.1	.744	58.2	...	...
100—125	212—257	4.7	9.8	.759	54.5	...	...
125—150	257—302	5.9	15.7	.789	47.4	...	...
150—175	302—347	5.9	21.6	.815	41.8	...	...
175—200	347—392	5.6	27.2	.837	37.3	...	...
200—225	392—437	6.3	33.5	.857	33.4	...	...
225—250	437—482	6.5	40.0	.887	27.8	...	...
250—275	482—527	8.0	48.0	.897	26.1	...	...
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	2.7	2.7	0.910	23.9	40	below 32
200—225	392—437	6.7	9.4	.924	21.5	50	41
225—250	437—482	8.1	17.5	.937	19.4	63	59
250—275	482—527	8.9	26.4	.951	17.2	108	78
275—300	527—572	10.4	36.8	.962	15.5	242	92

Residuum: Specific gravity, 1.003; Baume gravity, 0.5.*b*
Carbon residue, per cent, 14.1.
Residuum asphaltic.

a Third fraction from retort, 432 cc. or 12.2 per cent of total oil produced in run. See footnote to sample 305a.
b Baume heavy scale.

TABLE 26—Distillation analysis of shale-oil (continued).

Sample No. 305d.

Shale from Grand Valley, Colo.
Specific gravity oil, 0.922.
Viscosity at 130° F., 46.
Per cent tops, 42.2.
Per cent water, trace.

Retort: Horizontal rotary; fourth and last fraction of oil.*a*
Baume gravity, 21.8.
Setting point, 24° C. (75° F.).
Unsaturation tops, per cent, 46.3.
Index No. oil, 22.66.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 633 mm. First drop, 49° C. (120° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 130° F.	Setting point °F.
Up to 50	Up to 122	...	...	...	...	...	...
50— 75	122—167	1.4	1.4 }	0.731	61.5	...	...
75—100	167—212	1.7	3.1 }			...	...
100—125	212—257	4.1	7.2	.770	51.8	...	...
125—150	257—302	5.1	12.3	.792	46.8	...	...
150—175	302—347	5.5	17.8	.819	40.9	...	...
175—200	347—392	5.5	23.3	.844	35.9	...	...
200—225	392—437	6.1	29.4	.865	31.9	...	...
225—250	437—482	6.0	35.4	.886	28.0	...	...
250—275	482—527	6.8	42.2	.905	24.7	...	...
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	3.0	3.0	0.916	22.8	40	below 32
200—225	392—437	7.8	10.8	.935	19.7	49	41
225—250	437—482	8.5	19.3	.944	18.3	63	58
250—275	482—527	10.8	30.1	.966	14.9	121	82
275—300	527—572	9.0	39.1	.980	12.9	239	93

Residuum: Specific gravity, 1.021; Baume gravity, 3.0.*b*
Carbon residue, per cent, 13.5.
Residuum asphaltic.

---

a Fourth fraction from retort, 723 cc. or 20.2 per cent of total oil produced in run. See footnote to sample 305a.
b Baume heavy scale.

TABLE 26—Distillation analysis of shale-oil (continued).

Sample No. 306.

Shale from Grand Valley, Colo.
Specific gravity oil, 0.918.
Viscosity at 130° F., 53.
Per cent tops, 34.8.
Per cent water, trace.
Per cent sulphur in oil, 0.60.

Retort: Assay; rate oil production 1 cc. per minute.[a]
Baume gravity, 23.3.
Setting point, 22° C. (72° F.).
Unsaturation tops, per cent, 44.4.
Index No. oil, 19.35.
Per cent nitrogen in oil, 2.07.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 628 mm. First drop, 57° C (135° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 130° F.	Setting point °F.
Up to 50	Up to 122	...	...	...	...	...	...
50— 75	122—167	0.3	0.3	0.745	57.9	...	...
75—100	167—212	1.0	1.3				
100—125	212—257	2.2	3.5				
125—150	257—302	4.1	7.6	.782	49.0	...	...
150—175	302—347	4.6	12.2	.810	42.8	...	...
175—200	347—392	4.6	16.8	.837	37.3	...	...
200—225	392—437	5.4	22.2	.850	34.7	...	...
225—250	437—482	5.6	27.8	.870	30.9	...	...
250—275	482—527	7.0	34.8	.890	27.3	...	...
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	4.3	4.3	0.902	25.2	39	below 32
200—225	392—437	8.2	12.5	.920	22.2	48	45
225—250	437—482	10.7	23.2	.938	19.3	82	73
250—275	482—527	8.2	31.4	.953	16.9	148	90
275—300	527—572	11.3	42.7	.962	15.5	241	99

Residuum: Specific gravity, 0.992; Baume gravity, 11.1.
Carbon residue, per cent, 11.6.
Residuum asphaltic—softens at 35° C. (95° F.).

[a] Yield of crude oil from shale: 35.73 gallons per ton; scrubber naphtha, 1.17 gallons per ton; specific gravity scrubber naphtha, 0.714 (66.1° B.), unsaturation, 31.8%. Scrubber naphtha not included in calculating index number.

TABLE 26—Distillation analysis of shale-oil (continued).

Sample No. 307.

Shale from Grand Valley, Colo.
Specific gravity oil, 0.901.
Viscosity at 130° F., 50.
Per cent tops, 39.0.
Per cent water, trace.
Per cent sulphur in oil, 0.53.

Retort: Assay; rate of oil production, 0.2 cc. per minute.[a]
Baume gravity, 25.4.
Setting point, 26° C. (79° F.).
Unsaturation tops, per cent, 39.1.
Index No. oil, 23.75.
Per cent nitrogen in oil, 1.79.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 629 mm. First drop, 50° C. (122° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 130° F.	Setting point °F.
Up to 50	Up to 122	...	...	...	...	...	...
50—75	122—167	1.2	1.2	0.712	66.6	...	...
75—100	167—212	1.1	2.3			...	...
100—125	212—257	2.8	5.1	.750	56.7	...	...
125—150	257—302	4.4	9.5	.779	49.7	...	...
150—175	302—347	4.8	14.3	.803	44.4	...	...
175—200	347—392	5.3	19.6	.820	40.7	...	...
200—225	392—437	5.6	25.2	.846	35.5	...	...
225—250	437—482	6.4	31.6	.868	31.3	...	...
250—275	482—527	7.4	39.0	.885	28.2	...	...
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	4.3	4.3	0.895	26.4	41	below 32
200—225	392—437	9.4	13.7	.910	23.9	50	43
225—250	437—482	9.1	22.8	.928	20.9	70	65
250—275	482—527	10.4	33.2	.944	18.3	110	81
275—300	527—572	11.0	44.2	.955	16.6	190	95

Residuum: Specific gravity, 0.981; Baume gravity, 12.7.
Carbon residue, per cent, 6.1.
Residuum asphaltic, softens at about 40° C. (104° F.).

---

[a] Yield of crude oil from shale, 34.66 gallons per ton; scrubber naphtha, 0.80 gallons per ton. Not enough scrubber naphtha for unsaturation or gravity determinations. Scrubber naphtha not included in calculating index number.

TABLE 26—Distillation analysis of shale-oil (continued).

Sample No. 203a.

Shale from Soldier Summit, Utah.
Specific gravity oil, 0.922.
Viscosity at 140° F., 68.
Per cent tops, 24.79.
Per cent water, trace.

Retort: Laboratory vertical; steam used ratio 1:1; oil is composite of several runs.
Baume gravity, 21.9°.
Setting point, 37.5° C. (99° F.).
Unsaturation tops, per cent, 46.3.
Index No. oil, 13.30.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 637 mm. First drop, 55° C. (131° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 140° F.	Setting point °F.
Up to 50	Up to 122	...	...	...	...	...	...
50—75	122—167	0.67	0.67	0.791	47.0	...	...
75—100	167—212	0.67	1.34				
100—125	212—257	0.84	2.18				
125—150	257—302	0.84	3.02				
150—175	302—347	2.68	5.70	.814	42.0	...	...
175—200	347—392	2.68	8.38	.830	38.7	...	...
200—225	392—437	3.93	12.31	.840	36.7	...	...
225—250	437—482	5.53	17.84	.858	33.2	...	...
250—275	482—527	6.95	24.79	.869	31.1	...	...
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	1.16	1.16	.882	28.7	41	...
200—225	392—437	4.80	5.96				
225—250	437—482	5.84	11.80	.893	26.8	49	70
250—275	482—527	8.96	20.76	.928	20.9	58	88
275—300	527—572	5.77	26.53	.936	19.6	80	99

Residuum: Sp. gr., 0.991 (11.3° Be.); setting point.[a]

[a] Asphaltic; no definite setting point.

TABLE 26—Distillation analysis of shale-oil (continued).

Sample No. 203b.

Shale from Soldier Summit, Utah.
Specific gravity oil, 0.878.
Viscosity at 140° F., 89.
Per cent tops, 39.95.
Per cent water, nil.

This oil is part of Sample 203a, distilled to coke *once*.
Baume gravity, 29.5°.
Setting point, 24° C. (75° F.).
Unsaturation tops, per cent, 43.5.
Index No. oil, 22.59.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 639 mm. First drop, 30° C. (86° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 140° F.	Setting point °F.
Up to 50	Up to 122	0.75	0.75	0.738	59.7	...	...
50—75	122—167	0.96	1.71				
75—100	167—212	0.71	2.42				
100—125	212—257	1.93	4.35				
125—150	257—302	3.18	7.43	.780	49.5	...	...
150—175	302—347	5.30	10.73	.808	43.3	...	...
175—200	347—392	4.42	15.15	.820	40.7	...	...
200—225	392—437	6.70	21.85	.838	37.1	...	...
225—250	437—482	7.35	29.20	.853	34.1	...	...
250—275	482—527	10.75	39.95	.872	30.6	...	...
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	4.60	4.60	.881	28.9	39	...
200—225	392—437	8.70	13.30	.887	27.8	42	...
225—250	437—482	8.75	22.05	.897	26.1	48	64
250—275	482—527	9.50	31.55	.926	21.2	62	84
275—300	527—572	10.35	41.90	.936	19.6	70	96

Residuum: Sp. gr., 0.954 (16.8° Be.); setting point, 43° C. (109° F.).

TABLE 26—Distillation analysis of shale-oil (continued).

Sample No. 203c.

Shale from Soldier Summit, Utah.
Specific Gravity oil, 0.865.
Viscosity at 140° F., 35.
Per cent tops, 46.42.
Per cent water, nil.

This oil is part of sample 203a, distilled to coke *twice*.
Baume gravity, 31.8°.
Setting point, 20° C. (68° F.).
Unsaturation tops, per cent, 43.2.
Index No. oil, 26.37.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 637 mm. First drop, 37° C. (99° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 140° F.	Setting point °F.
Up to 50	Up to 122	0.33	0.33	0.743	58.4	...	...
50—75	122—167	2.33	2.66				
75—100	167—212	1.93	4.59				
100—125	212—257	1.93	6.52				
125—150	257—302	3.00	9.52	.798	45.4	...	...
150—175	302—347	4.67	14.19	.805	43.9	...	...
175—200	347—392	5.26	19.45	.823	40.1	...	...
200—225	392—437	7.44	26.89	.840	36.7	...	...
225—250	437—482	9.36	36.25	.851	34.5	...	...
250—275	482—527	9.17	46.42	.870	30.9	...	.
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	5.60	5.60	.880	29.1	37	...
200—225	392—437	9.40	15.00	.886	28.0	39	...
225—250	437—482	15.20	30.20	.901	25.4	44	68
250—275	482—527	13.05	43.25	.920	22.2	55	82
275—300	527—572	7.30	50.55	.932	20.2	77	103

Residuum: Sp. gr., 0.968; (14.6° Be.). Setting point.[a]

[a] Asphaltic; no definite setting point.

TABLE 26—Distillation analysis of shale-oil (continued).

Sample No. 214.

Shale from Soldier Summit, Utah.
Specific gravity oil, 0.908.
Viscosity at 130° F., 45.
Per cent tops, 34.2.
Per cent water, trace.

Retort: Assay; rate of oil production, 1 cc. per minute.[a]
Baume gravity, 24.2.
Setting point, 30° C. (86° F.).
Unsaturation tops, per cent, 40.5.
Index No. oil, 20.34.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 630 mm. First drop, 55° C. (131° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 130° F.	Setting point °F.
Up to 50	Up to 122	...	...	...	...	...	...
50—75	122—167	0.6	0.6	0.741	58.9	...	...
75—100	167—212	1.3	1.9			...	...
100—125	212—257	2.4	4.3	.777	50.2	...	...
125—150	257—302	3.2	7.5	.791	47.0	...	...
150—175	302—347	4.5	12.0	.817	41.4	...	...
175—200	347—392	4.4	16.4	.830	38.7	...	...
200—225	392—437	5.2	21.6	.845	35.7	...	...
225—250	437—482	5.6	27.2	.863	32.2	...	...
250—275	482—527	7.0	34.2	.879	29.3	...	...
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	3.6	3.6	0.880	29.1	38	33
200—225	392—437	9.1	12.7	.900	25.6	42	54
225—250	437—482	8.5	21.2	.920	22.2	48	73
250—275	482—527	8.5	29.7	.938	19.3	62	86
275—300	527—572	12.9	42.6	.956	16.4	113	102

Residuum: Specific gravity, 0.990; Baume gravity, 11.4.
Carbon residue, per cent, 9.0.
Residuum, waxy; setting point not determined.

---

[a] Yield of crude oil from shale, 49.5 gallons per ton; scrubber naphtha, 1.11 gallons per ton. Not enough scrubber naphtha for unsaturation and gravity tests. Scrubber naphtha not included in calculating index number.

TABLE 26—*Distillation analysis of shale-oil (continued).*

Sample No. 215.

Shale from Soldier Summit, Utah.
Specific gravity oil, 0.890.
Viscosity at 130° F., 42.
Per cent tops, 38.5.
Per cent water, none.

Retort: Assay; rate of oil production, 0.2 cc. per minute.*a*
Baume gravity, 27.3.
Setting point, 26.5° C. (80° F.).
Unsaturation tops, per cent, 39.6.
Index No. oil, 23.25.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 634 mm. First drop, 53° C. (127° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 130° F.	Setting point °F.
Up to 50	Up to 122	...	...	...	...	...	...
50— 75	122—167	0.9	0.9	0.735	60.5	...	...
75—100	167—212	1.4	2.3			...	...
100—125	212—257	3.3	5.6	.774	50.9	...	...
125—150	257—302	4.3	9.9	.792	46.8	...	...
150—175	302—347	4.4	14.3	.809	43.1	...	...
175—200	347—392	4.7	19.0	.830	38.7	...	...
200—225	392—437	5.4	24.4	.845	35.7	...	...
225—250	437—482	6.6	31.0	.862	32.4	...	...
250—275	482—527	7.5	38.5	.878	29.5	...	...
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	5.5	5.5	0.890	27.3	38	below 32
200—225	392—437	9.2	14.7	.902	25.2	45	43
225—250	437—482	9.9	24.6	.920	22.2	51	72
250—275	482—527	12.2	36.8	.941	18.8	65	88
275—300	527—572	10.2	47.0	.952	17.1	104	103

Residuum: Specific gravity, 0.977; Baume gravity, 13.3.
Carbon residue, per cent, 6.4.
Residuum, waxy; setting point, 43.5° C. (110° F.).

a Yield of crude oil from shale, 46.61 gallons per ton; scrubber naphtha, 1.32 gallons per ton. Scrubber naphtha not included in calculating index number.

TABLE 26—Distillation analysis of shale-oil (continued).

Sample No. 801.

Shale from Indiana.
Specific gravity oil, 0.940.
Viscosity at 130° F., 43.
Per cent tops, 47.5.
Per cent water, trace.

Retort: Assay; oil is a mixture from several miscellaneous samples; produced at rate 1cc. per min.
Baume gravity, 18.9.
Setting point, below 0° C. (32° F.).
Unsaturation tops, per cent, 37.7.
Index No. oil, 29.59.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 622 mm. First drop, 42° C. (108° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 130° F.	Setting point °F.
Up to 50	Up to 122	0.7	0.7	0.707	68.0	...	...
50—75	122—167	1.6	2.3				
75—100	167—212	2.6	4.9	.743	58.4	...	...
100—125	212—257	4.6	9.5	.768	52.3	...	...
125—150	257—302	4.7	14.2	.795	46.1	...	...
150—175	302—347	5.7	19.9	.821	40.5	...	...
175—200	347—392	6.2	26.1	.847	35.3	...	...
200—225	392—437	6.7	32.8	.871	30.7	...	...
225—250	437—482	6.6	39.4	.896	26.3	...	...
250—275	482—527	8.1	47.5	.920	22.2	...	...
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	2.1	2.1	0.932	20.2	40	below 32
200—225	392—437	9.2	11.3	.960	15.8	52	below 32
225—250	437—482	5.4	16.7	.982	12.6	95	below 32
250—275	482—527	8.3	25.0	1.001	0.2a	230	45
275—300	527—572	8.6	33.6	1.019	2.7a	952	67

Residuum: Specific gravity, 1.062; Baume gravity, 8.5.a
Carbon residue, per cent, 22.0.
Residuum, asphaltic.

a Baume heavy scale.

TABLE 26—Distillation analysis of shale-oil (continued).

Sample No. 101.

Shale from Scotland.
Specific gravity oil, 0.864.
Viscosity at 140° F., 41.
Per cent tops, 37.10.
Per cent water, 0.62.

Retorted in Bureau of Mines.
Assay retort, 2 hrs. retorting time.
Baume gravity, 32°.
Setting point, 32° C. (90° F.).
Unsaturation tops, per cent, 32.0.
Index No. oil, 25.21.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 647 mm. First drop, 44° C. (111° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Unsaturation per cent	Viscosity at 140° F.	Setting point °F.
Up to 50	Up to 122	0.169	0.169					
50— 75	122—167	0.236	0.405					
75—100	167—212	1.18	1.42	.740	59.2	...	...	...
100—125	212—257	2.77	4.19					
125—150	257—302	4.06	8.25	.770	51.8	...	...	...
150—175	302—347	3.82	12.07	.798	45.4	...	...	...
175—200	347—392	4.56	16.63	.810	42.8	...	...	...
200—225	392—437	5.57	22.20	.825	39.7	...	...	...
225—250	437—482	7.33	29.53	.840	36.7	...	...	...
250—275	482—527	7.57	37.10	.855	33.7	...	...	...
Vacuum distillation at 40 mm.								
Up to 200	Up to 392	2.10	2.10	.868	31.3	...	38	...
200—225	392—437	4.38	6.48	.872	30.6	...	41	...
225—250	437—482	5.88	12.36	.874	30.2	...	43	70
250—275	482—527	7.30	19.66	.894	26.6	...	50	84
275—300	527—572	9.45	29.11	.898	25.9	...	60	99

Residuum: Sp. gr., 0.968 (14.6° Be.); setting point, 45° C. (113° F.).

TABLE 26—Distillation analysis of shale-oil (continued).

Sample No. 102.

Crude shale oil from Scotland.[a]
Specific gravity oil, 0.877.
Viscosity at 140° F., 43.6.
Per cent tops, 35.44.
Per cent water, 0.13.

Baume gravity, 29.6.
Setting point, 28° C. (82.4° F.).
Unsaturation tops, per cent, 31.9.
Index No. oil, 24.16.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 644 mm. First drop, 49° C. (120° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 140° F.	Setting point °F.
Up to 50	Up to 122	tr.	...	...	...	...	...
50— 75	122—167	0.13	0.13				
75—100	167—212	0.32	0.45				
100—125	212—257	0.99	1.44	0.760	54.2	...	...
125—150	257—302	1.66	3.10				
150—175	302—347	3.00	6.10	.785	48.3	...	...
175—200	347—392	6.12	12.22	.807	43.5	...	...
200—225	392—437	8.10	20.32	.826	39.5	...	...
225—250	437—482	6.65	26.97	.842	36.3	...	...
250—275	482—527	8.47	35.44	.857	33.4	...	...
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	9.32	9.32	.871	30.6	38	...
200—225	392—437	5.27	14.59	.881	28.9	41	...
225—250	437—482	7.16	21.75	.892	27.0	46	76
250—275	482—527	6.13	27.88	.902	25.2	52	82
275—300	527—572	6.07	33.95	.911	23.7	60	93

Residuum: Sp. gr., 0.957 (16.3° Be.); setting point, 41° C. (106° F.).

[a] Represents approximately 90 per cent of the oil produced from the shale. The remainder, 10 per cent, is recovered as scrubber naphtha and is kept separate from the crude in refining. If the proper proportion of scubber naphtha were added to the crude, the specific gravity of the mixture would be about 0.860, and the index number much higher.

TABLE 26—Distillation analysis of Pennsylvania crude petroleum (concluded).

Sample No. 1107.

Specific gravity, 0.812.
Viscosity at 140° F., 38.8.
Per cent tops, 52.76.
Per cent water, trace.
Baume gravity, 42.4.
Setting point.[a]
Unsaturation tops, per cent, 4.1.
Index No. oil, 50.60.

Distillation, Bureau of Mines, Hempel Method.

Air distillation: Barometer, 644 mm.; first drop, 26° C. (79° F.).

Temperature °C.	Temperature °F.	Per cent cut	Sum per cent	Sp. Gr. cut	Degrees Baume cut	Viscosity at 140° F.	Setting point °F.
Up to 50	Up to 122	0.90	0.90	0.674	77.7	...	...
50—75	122—167	1.61	2.51				
75—100	167—212	4.08	6.59	.712	66.6	...	...
100—125	212—257	8.29	14.88	.733	61.0	...	...
125—150	257—302	5.46	20.34	.752	56.2	...	...
150—175	302—347	6.77	26.11	.763	53.5	...	...
175—200	347—392	5.82	31.93	.778	50.0	...	...
200—225	392—437	6.95	38.88	.789	47.4	...	...
225—250	437—482	6.42	45.30	.800	45.0	...	...
250—275	482—527	7.46	52.76	.812	42.4	...	...
Vacuum distillation at 40 mm.							
Up to 200	Up to 392	3.33	3.33	.826	39.5	39	...
200—225	392—437	7.75	11.08	.832	38.3	40	...
225—250	437—482	6.02	17.10	.841	36.5	45	60
250—275	482—527	5.37	22.47	.848	35.1	51	72
275—300	527—572	5.16	27.63	.859	33.0	67	86

Residuum: Sp. gr., 0.882 (28.7° Be.); setting point, 18° C. (65° F.).

[a] Not determined.

PART IV—CONCLUSIONS BASED ON EXPERIMENTAL RETORTING OF OIL-SHALES.

Below are summarized conclusions based on the experimental retorting of oil-shales by the Bureau of Mines in its laboratories at Salt Lake City, Utah (in co-operation with the Department of Metallurgical Research of the University of Utah), and Boulder, Colo. (in co-operation with the State of Colorado).

FRACTIONAL "EDUCTION".

When an oil-shale is distilled it does not first yield a light oil, and as the temperature increases, heavier oils, as does petroleum when subjected to increasing temperatures. Although there is a variation in the quality of oil produced during the course of retorting, it is not of the nature of fractional distillation, nor does it appear to be of such nature that it can be taken advantage of in commercial work. There is no evidence to support the theory of fractional eduction. (See page 46.) Analyses of samples 305a to 305d, Table 26, which were taken at different stages of retorting experiment, show very clearly the nature of the variation referred to.

INFLUENCE OF RATE OF RETORTING.

Other conditions being constant, rapid retorting—rapid heating and rapid production of oil—is favorable for high yields of oil; slow retorting gives a smaller yield, but the oil recovered is of better quality. This is shown in Table 27 below, and in greater detail by analyses of samples 214 and 215, and 306 and 307, Table 26.

TABLE 27—Summary of retorting experiments on oil-shale from Soldier Summit, Utah. Assay retorts used. a

Time of retorting, hours *b*	2	4	6	8	10	20
Oil yield, gallons per ton	44.6	43.0	42.2	41.9	41.7	41.1
Specific gravity of oil	0.891	0.884	0.879	0.876	0.875	0.872
Melting point of oil °C.	34.0	30.3	30.0	29.8	29.6	28.5
Tops to 275°C. per cent	33.9	36.4	37.8	38.2	38.3	39.0
Saturation of tops, per cent	61.0	63.8	65.0	65.3	65.7	67.3
Index number *c*	9.2	10.0	10.3	10.5	10.6	10.8
Temperature to finish distillation °C *d*	526	467	458	454	452	449

a Experimental work by L. C. Karrick, Bureau of Mines laboratory, Salt Lake City, Utah.

b This is elapsed time from beginning to end of oil production. In all cases shales were completely retorted, i. e., they would yield no more oil if heated longer or to a higher temperature. Oil-production rate constant during each test.

c This is second index discussed on page 163. It is based on yield of crude oil, percentage of tops, and saturation of tops.

d This is the temperature at which last oil distilled from shale.

Particularly noteworthy in the above table is the data on temperature required to complete distillation. It is seen that if the shale is retorted rapidly, oil production is completed at a higher temperature than if the oil is produced slowly. These results were obtained on finely-ground shale (minus ¼ inch). If larger pieces were used, it is probable that even higher temperatures would be required to complete distillation in the same time.

Other results of rapid retorting, as compared with slow retorting, are: A slightly smaller percentage of spent shale; a smaller amount of fixed carbon in the spent shale; a larger amount of solid paraffin in the oil; and a larger amount of nitrogen in the oil, and less in the spent shale.[127] As regards the relative amounts of nitrogen in spent shale and oil produced at different rates of retorting, the differences indicated as percentages in oil and spent shale are not particularly striking, but if calculated to actual weights of nitrogen in these two products, especially when it is considered that a greater weight of spent shale and smaller weight of oil is produced by slow retorting, the differences indicate that rate of retorting will have an important influence on amount of ammonia producible from the spent shale, and the amount of basic nitrogen compounds contained in the crude oil. Oils produced by slow retorting appear to contain less sulphur than those produced more rapidly, but more experimental work is required to establish definitely the effect of rate of retorting on distribution of sulphur.

It has been argued that in these experiments factors other than rate of heating or oil production were varying. This argument is sound, since when the oil is produced slowly, velocity of vapors through and from the retort is lower, the average temperature to which the vapors are exposed is lower, and the degree of condensation and redistillation undoubtedly is different than when a rapid rate is used. However, these are all dependent on the rate of heating, and there appears to be no practical means of altering them in any given retort except by using steam or a scavenging gas. As is shown below, the effect of passing steam or non-oxidizing gases through the retort, at any given rate of oil production, is to produce an oil having the characteristics of one produced at a more rapid rate without the use of scavanging agents. The writer and his associates believe that slow distillation permits more complete conversion of the first-formed bitumen (see page 44) into desirable oils, and when the shale is retorted slowly, that this conversion takes place at a lower temperature than under conditions of rapid retorting. They also

127 This apparently contradicts results obtained on Scotch shale by Beilby. See Beilby, G. T., Production of ammonia from the nitrogen of minerals: Jour. Soc. Chem. Ind., vol. 3, 1884, pp. 216-224; Jour. Roy. Soc. Arts, vol. 33, 1885, pp. 313-320. The writer and his associates, however, have reached the above conclusions as the results of work on representative American shales and a typical Scotch shale.

believe that in the experiments performed, possible differences in degree of condensation and redistillation, and temperatures and time during which the vapors were exposed to these temperatures after formation, had relatively little to do with the amount and quality of oil produced. In larger retorts, however, where there would be longer time of contact of vapors with hot retort walls, longer exposure to possibly higher average temperatures, and probability of considerable refluxing condensation and redistillation on account of greater differences in temperature between different parts of the distilling shale mass, these factors may have important influences. The above conclusions have resulted from work on several different types of small laboratory retorts.

INFLUENCE OF STEAM OR OTHER GASES.

Passing steam or other non-oxidizing gases through the retort —all other conditions being constant—produces an effect similar to a more rapid rate of retorting without using these gases. That is, with any given retort, and at any given rate of oil production, the oil produced with steam will have more nearly the properties of an oil made at a more rapid retorting rate without steam. The writer believes that in large retorts, particularly of the vertical type, for reasons given in the preceding section, the quality and amount of oil recovered would be relatively low, unless the retorts were steamed.

Steam in a retort adds volume to the vapors and thereby gives them a higher velocity, sweeping them from the retort so rapidly that reflux condensation is reduced to a minimum, and time of exposure to high temperatures is very short. Mention has been made that the first effect of heat on the kerogen of the shale is to convert the kerogen into a bitumen. Undoubtedly this bitumen is fluid at temperatures at which it is formed, and therefore it is probably held in the pores and on the surfaces of the shale lumps. If the temperature and rate of distillation is high, the bitumen will rapidly decompose into oil vapors and gas. These will evolve rapidly from the shale and carry with them a certain amount of undecomposed bitumen, producing an oil containing a relatively large amount of undecomposed bitumen. This oil will be relatively large in quantity, poor in quality, and contain a relatively large percentage of nitrogen. The spent shale from such a distillation will contain relatively small amounts of fixed carbon and nitrogen. If, at any rate of retorting, steam or other scavenging gas is passed through the retort, the effect on the undecomposed bitumen will be that of a steam distillation, and more undecomposed bitumen will be distilled than if steam were not used. The steam will protect the oil vapors and undecomposed bitumen—in vapor form and mechanically carried—from further decomposition in passing through the retort.

If, however, the shale is distilled slowly—at a slowly rising or even practically uniform temperature—conversion of kerogen into bitumen will take place slowly, and decomposition of the latter will go on slowly and at relatively low temperature; conditions favorable for the production of desirable oil-hydrocarbons, though the absolute yield of crude oil will be smaller than when retorting is rapid. Thus, with slow retorting, decomposition of bitumen to desirable products will be more complete, and there will be less carrying over, or distillation, of undecomposed or partly undecomposed bitumen. Possibly the bitumen produced at low temperatures will yield better products than those yielded by bitumens formed at somewhat higher temperatures under conditions of rapid oil production.

Thus the net result of slow distillation would be a somewhat smaller amount of oil than would be produced by rapid distillation, but the oil would be of better quality, except for amount of solid paraffins; the spent shale would contain a smaller amount of fixed carbon; and since when the bitumen decomposes it appears to leave much of its nitrogen in the resultant coke, the oil would contain less, and the spent shale more, nitrogen.

In certain types of retorts, however, particularly those that are vertical, the vapors might be still more completely decomposed into gases and undesirable hydrocarbons, as mentioned before. Therefore the use of steam would be necessary, and if production and conversion of the bitumen were slow enough, and took place at low enough temperature, the amount of bitumen steam-distilled would probably not be enough materially to lower the quality of the resulting oil. In any event, when any definite amount of steam is used, the quality of the recovered oil improves as rate of distillation decreases, just as it does when no steam is used, although as before mentioned, for any particular rate of retorting with the small experimental retorts thus far used, better oil is produced when steam is not used, and its quality decreases as the amount of steam is increased. At the same time, the quantity recovered increases in relatively the same proportion.

The rate of distillation chosen, and the amount of steam used will thus depend on the type and size of retort; the nature of oil desired; whether nitrogen is wanted in the oil, or in the spent shale for ammonia production; the use to which the spent shale is to be put—that is, is it to be used in making water-gas for fuel: if so the higher the percentage of fixed carbon in it, the better—; and how much the quality of the crude oil can be improved by subsequent refinery operations. (See page 187.) The proper combination will be determined largely by the cost sheet, and will be that which will yield the largest profit per unit of shale treated.

If these conclusions are correct, and there is much experimental evidence to confirm them,[128] it becomes clear why the Scotch type of retort is designed to produce oil slowly. Not only is a better oil produced than if retorting were rapid; but more ammonia can be made from the spent shale and protected from decomposition by rapid removal from the retort; there is a lower percentage of nitrogen in the oil, and consequently a smaller amount of nitrogen compounds to remove in refining; and there is more fuel value in the spent shale, from which fuel-gas is made. Heat is recovered from the spent shale and put back into the distilling zone; heat distribution is improved; and the oil vapors are swept from the retort before undue decomposition can take place. The capacity of a Scotch retort, calculated in terms of tons of shale or gallons of oil, is low, but for Scotch shales and conditions, it is such that it probably yields the greatest profit. Undoubtedly if desired, the daily amount of shale put through could be increased materially over that now treated per day. The capacity of any shale retort, up to a point determined largely by mechanical limitations, is, or should be, governed by the conditions that yield the greatest profit.

INFLUENCE OF SIZE OF SHALE RETORTED.

Other conditions being constant, size of shale pieces retorted, of itself, has little or no effect on the quantity and quality of the crude oil produced. If anything, the larger sizes yield a little less oil of somewhat better quality than do the smaller. However, when a piece of shale is broken or crushed, and various sizes separated by screening, the larger sizes yield more and better oil than the smaller. Thus, a Colorado shale was crushed, and separated into four sizes by screening. The largest size (plus 1 inch) yielded at the rate of about 38 gallons of oil per ton, while the smallest (minus 10 mesh) yielded but 27 gallons. Though the rate of retorting was the same with both samples, the finer shale yielded oil of much lower quality.

If, however, large pieces of shale are crushed to small size, and all of the crushed material retorted, the yield and quality of oil will be practically the same as can be produced at the same rate of retorting from the larger pieces. In other words, when a shale is crushed, the leaner parts break more readily than the richer. For this reason it may sometimes be advantageous to pay no attention to fines produced in mining or crushing, especially if the retort used is particularly adapted to treat shale in large lumps. Possibly the fine material will be used directly as fuel, or gasified in producers.

---

128 A technical paper is now being prepared which gives full details of the experimental evidence on which these conclusions are based.

INFLUENCE OF TYPE OF RETORT.

It is obvious from the discussion on page 182 that the design and method of operation of the retort has an important effect on the amount and quality of products made from any oil-shale. A large amount of experimental work must be done before the effects of many factors of retort design and operation can be fully determined, but results already obtained show clearly that merely by altering retort design or retorting conditions slightly the character of products obtained from a given shale can be materially changed. In large commercial retorts, the character of recovered products will largely be determined by what happens to the vapors after they are produced from the shale. Exposure of vapors to excessive temperatures for any considerable time or contact with hot metallic surfaces is certain to have a harmful effect.

QUALITY OF OIL PRODUCED FROM DIFFERENT SHALES.

As noted on page 33, different shales retorted under identical conditions yield oils of different quality. This is more fully brought out by analyses of samples 214, 306, 101, and 801, Table 26. These oils were produced from different shales, as indicated in the table, but all were made under identical retorting conditions.

COKING DISTILLATION OF SHALE-OILS.

In the experimental studies just discussed, it was noted that the highest yields of oils were produced from oil-shales by rapid distillation, distillation with steam, or by a combination of both methods, but that in such cases the quality of the recovered oils was lower than that of oils produced by slow distillation, or without steam. The results obtained in the early stages of this investigation, considering the nature of the retorts used, led to the belief—since abandoned—that possibly the beneficial effect of the slow distillation was due to the condensation and gentle distillation to coke, within the retort itself, of the oil first produced from the shale. This suggested that the oil produced when highest recoveries were obtained might be re-distilled to coke in an effort to improve the quality of the oil. Results of such redistillation are shown in Table 28, and the characteristics of the oils produced in one and two coking distillations are shown in Table 26 (Samples 203a, 203b, and 203c).

TABLE 28—Effect of one and two coking distillations on crude shale oil.

Sample No.	Oil	Sp. Gr. oil	Setting point °F	Per cent tops	Unsaturation tops	Index No. oil	Sp. Gr. residuum	Nature residuum
203*a*	Utah crude shale oil produced with steam	0.922	99	24.79	46.3%	13.30	0.991	Asphaltic
203*b*	Same—once run to coke	0.878	75	39.95	43.5	22.59*a*	0.954	Waxy S.P. 109° F.
203*c*	Same—twice run to coke	0.865	68	46.42	43.2	26.37*a*	0.968	Asphaltic

a In comparing these oils consideration must be given to the quantity lost in the coking distillations. (See page 187.)

It is interesting to note that the results of a single distillation to coke, at atmospheric pressure were, first the production of a large amount of still coke; second, increase in the size of the motor fuel fraction; third, a decrease in the amount of unsaturates in this fraction; and fourth, an evident loss of solid paraffin wax, as indicated by the setting points of the vacuum fractions. A second re-distillation produced only a small amount of coke and the changes in the recovered oil were not so great, but were in the same direction as those produced by the first distillation.

The first step in refining shale oil commercially in Scotland and Australia, is to run the total crude from the retort condensers down to coke, before fractionation and chemical treatments are begun. This procedure is said to facilitate the subsequent refining operations and to improve the quality of finished products over those obtained from a crude not so handled.

There are several reasons why a coking distillation might improve the crude shale oils in the directions noted. The matter has been discussed by several investigators, many of whom are inclined to consider the phenomena as something new and mysterious. In the opinion of the writer there is little mysterious in the process, and certainly nothing novel, as it is common practice in Scotland.

When the oil is distilled to coke, many of the heavier unsaturated compounds in the oil probably break down, producing coke, or carbon, and lighter unsaturated and saturated hydrocarbons. Consequently, the percentage of light oils in the recovered distillate would be increased, and the total percentage of unsaturates in that fraction would be increased, as the saturated oils originally in that fraction would be undecomposed, and to them would be added the light saturated and unsaturated oils produced by coking. Possibly, also, some of the heavier paraffins would break down to lighter paraffins and unsaturated bodies, thus accounting for the decreased content of paraffin wax.

Botkin[129] believes that complex nitrogen bodies in the oil, possibly undecomposed bitumen, break down on redistillation of this nature, yielding a distillate with a smaller percentage of nitrogen, most of the nitrogen remaining in the coke. As the percentage of nitrogen bases in the distillate would be smaller, there would be a smaller loss on treatment with sulphuric acid, and consequently a lower "unsaturation". (One should clearly understand here what is meant by "unsaturation", or sulphuric acid absorption. See page 162.) There is a good deal of experimental evidence to support this belief.

RE-DISTILLATION OF SHALE-OILS FROM UTAH SHALE.

A composite sample of shale-oil (Sample 203a, Table 26) representing several runs under different conditions was used in the experimental work. The oil had been produced from oil-shale from Soldier Summit, Utah, and its quality was somewhat inferior to that usually produced from these shales by the Bureau. Since then, many similar tests with similar results have been made on this and other oils.

This oil was sampled and an analytical distillation made. The balance of the sample was cut in two portions, the first of which was distilled to coke in a copper still. The results of this distillation are as follows:

	Per cent
Oil used	100.0
Oil recovered	87.5
Coke produced	9.2
Lost as gas	3.3

The recovered distillate or "**once run crude**" was subjected to analytical distillation. (Sample 203b, Table 26.)

The second half of the original crude sample was then run to coke as above, with the following results:

	Per cent
Oil used	100.0
Oil recovered	87.0
Coke produced	9.1
Lost as gas	3.9

The oil recovered in this distillation, "**once run oil**", was then placed in another copper-coking still, and immediately run to coke a second time, producing Sample 203c, "**twice run oil**".

The results of this second coking distillation are:

Oil used, on basis "once run oil", 100.0%. On basis original crude, 87%.

Oil recovered, on basis "once run oil", 98.1%. On basis original crude, 85.3%.

129 Botkin, C. W., A study of the saturated and unsaturated oils from shale: Chem. and Met. Eng., vol. 26, No. 9, March 1, 1922, pp. 398-401.

Coke produced, on basis "once run oil", 1.6%.

Lost on gas, on basis "once run oil", 0.3%.

It will be noted from Table 28 that the index numbers of the oils used in the re-distillation experiments are:

Crude oil	13.30
Once run oil	22.59
Twice run oil	26.37

To obtain a true idea as to the results of the coking distillations, these indices must be corrected for the losses incurred in the re-distillations. 12.5 per cent was lost as gas and coke in the first distillation, and 13.0 plus 1.9 in producing the twice run oil.

Correcting the index values given above for these losses, the numbers referred to the quality and quantity of the original crude, become:

Crude oil	13.30
Once run oil	19.77
Twice run oil	22.42

The effect of a coking distillation is of great importance in retort design and operation and deserves careful consideration. A coking distillation improves the quality of an oil for refining, and the oil will yield a larger net amount of desirable refined products, not only because the preliminary coking distillation will increase the percentage of light distillates, but also because there will be a smaller loss in treating them. To offset this is the loss through production of coke and gas. Another manner of obtaining an oil of good quality is to produce it more slowly from the shale, and this oil in turn can be further improved by coking it. The effects of re-distillations, or coking distillations, can be regulated by the method of making the distillation, that is, by rate, degree of refluxing condensates, and use of steam, pressure or vacuum.

In the opinion of the writer, the full application of the effects of coking distillations may result in the design of retorts to produce the oil from shale as rapidly as possible, and with the use of steam, especially if no attempt is to be made to produce ammonia compounds. This will result in maximum oil yield and its quality can be adjusted within fairly wide limits by suitable coking distillation. Slower retorting would produce results somewhat similar, but rather than reduce the capacity of expensive retorts for the sake of quality of oil, it will probably be cheaper, and lead to more economical final results, if the retorts are run at capacity and relatively inexpensive coking-stills are used for the production of quality. In effect this will be the same as reducing the investment in retorting equipment per barrel of oil produced. Stills will probably be cheaper than retorts and can

more readily be controlled. An economic balance will probably be found in the rate of oil production and method of redistillation which will lead to the highest economic returns. The by-product of coking distillations—coke—should be easily marketed.

NATURE OF SHALE GAS.

The fixed gases produced when different shales are distilled naturally differ in volume and composition. Likewise the amount and composition of gas produced from a given shale differ with the conditions of retorting the shale. Evolution of gas begins at a temperature below that of first oil production and continues during the oil-producing period. After all the oil has been produced, the evolution of gas drops off sharply and its heating value decreases, although if heating continues, the shale will give off gas for a long time after oil production has ceased. The first gas produced is often rich in hydrogen sulphide, but the evolution of this undesirable constituent drops off as the temperature of the shale increases. With some shales, however, as much as 20 per cent of the total gas produced by dry distillation is hydrogen sulphide, and all shale gases thus far examined have contained relatively large amounts of this substance.

Investigational work has not progressed far enough to permit reliable conclusions to be drawn regarding the amount of gas produced from shales of different richness, or the change in gas composition caused by different retorting conditions. The Bureau of Mines has thus far worked only with gas produced by dry distillation, and has not yet examined gases produced from retorts that are steamed during distillation and in which some of the steam reacts with the carbon of the spent shale, producing a rather low-grade water gas. This gas mixes with the gases of destructive distillation produced in another part of the retort. A large volume of gas of low heating value is produced from the relatively lean Scotch shales as a result of steaming in this manner. The composition and heating value of Scotch shale gas is shown on page 77. In Table 29, below, is shown the amount, composition and heating value of gases produced from two typical American shales. Sample No. 1 was produced from DeBeque, Colo., shale during the retorting test in which oil sample No. 306 (Table 26) was collected; and sample No. 2 was made from Soldier Summit, Utah, shale when oil sample No. 214 was produced. Oil-production rate was constant and identical in each case, and the retorts were heated to approximately the same final temperature for thirty minutes after oil production had ceased. Sample No. 3 was also produced from DeBeque shale (the same shale used in producing Sample No. 1), but the shale was retorted in a horizontal rotary retort used in the Boulder laboratory. The rate of oil production was comparable with

that used when samples 1 and 2 were collected, and likewise the shale was heated to about the same final temperature for 30 minutes after oil production stopped. The gas samples were scrubbed to remove hydrogen sulphide before analyses were made, and results are reported on hydrogen sulphide and air-free basis. The hydrogen sulphide percentages shown in parentheses were determined on gas samples drawn directly from the gasometers before the gas was passed through scrubbers to remove hydrogen sulphide prior to complete analysis.

TABLE 29—Nature and composition of typical oil-shale gases produced by dry distillation.

Retort Used	Assay	Assay	Horizontal rotary
Gas sample No.	1	2	3
Shale	DeBeque, Lot 2	Soldier Summit	DeBeque, Lot 2
Volume gas per ton of shale, cubic feet *a*	787	1219	1120
Gross heating value per cubic foot *a*	807	765	1079
Analysis	Per cent	Per cent	Per cent
Carbon dioxide (CO_2)	17.1	11.0	13.8
Illuminants (C_nH_{2n})	6.4	3.1	11.6
Carbon monoxide (CO) ...	6.4	3.9	4.4
Oxygen (O_2)	0.0	0.0	0.0
Hydrogen (H_2)	35.8	47.5	30.2
Methane (CH_4)	21.2	25.8	23.5
Ethane (C_2H_6)	12.3	8.7	16.5
Nitrogen (N_2)	0.8	0.0	0.0
Total	100.0	100.0	100.0
Hydrogen sulphide	(1.4)	(6.1)	(1.3)

a At O°C., and 760 mm. pressure. Heating value determined with Junker's calorimeter.

SELECTED BIBLIOGRAPHY.

Compiled by Miss E. H. Burroughs.

Selected by Martin J. Gavin.

The following bibliography has been made as short as possible, consistent with the presentation of literature that covers oil-shale from all angles. References to many articles have been omitted purposely because the material in them is duplicated by publications included in the bibliography. An attempt has been made to eliminate articles evidently published for promotion or propaganda purposes.

The Bureau of Mines has published, as Reports of Investigations, Serial No. 2277, a selected bibliography of oil-shales, prepared by E. H. Burroughs and M. J. Gavin, and issued September, 1921. This bibliography lists the more noteworthy articles on oil-shale that have appeared in the literature of the years 1915-1920, together with references to standard publications that appeared previous to 1915. A total of 295 references is listed, and although it by no means intends to be a complete bibliography of the subject, in the opinion of the writer it contains references to the most important articles. From time to time this bibliography will be revised and brought up-to-date. Copies may be obtained on application to the Director, Bureau of Mines, Washington, D. C. The applicant should request the most recent revision of the Bibliography of Oil-Shale.

ABRAHAM, HERBERT. Asphalts and allied substances. Their occurrence, modes of production, uses in the arts, and methods of testing. New York, Van Nostrand, 2nd ed., 1920. 608 pp. Includes chapters on pyrobituminous shales (pp. 158-164), giving their occurrence and properties; and on shale-tar and shale-tar pitch (pp. 216-224); giving description, distillation methods and products.

ALDERSON, V. C. The oil-shale industry. New York, Frederick A. Stokes Co., 1920. 175 pp. Chapters on the nature, origin and distribution of oil-shale, the history of oil-shale, including Scotch and American investigations, mining of oil-shale, retorting and reduction, experimental and research work, economic factors, a summary of important features of the industry, opinions of authorities on the subject, and discussion of the future of the industry. Concludes with a selected bibliography.

ALDERSON, V. C. The oil shale industry in Scotland and England. Colorado School of Mines Quarterly, vol. 15, Oct., 1920. 28 pp. Used also as address before the Independent Oil Men's Association, September, 1920, and published in somewhat abbreviated form in the Railroad Red Book, vol. 37, Oct., 1920, pp. 891-893, 895, 897-899, 902-905. Discusses, under Scotland, the mode of occurrence, mining, properties, and retorting of the shale and refining of its products; under England, mode of occurrence, analysis and properties of the shale, and removal of sulphur. Includes directory of Scottish and English oil shale retorts. Also in Am. Gas Eng. Jour., vol. 113, Nov. 14, 1920, pp. 255-260. Shale Rev., vol. 2, Oct., 1920, pp. 5-7; Nov., pp. 12-14. Petroleum Annual, 1921, Independent Oil Men's Association, Chicago, pp. 269-279. Min. and Oil Bull., vol. 7, March, 1921, pp. 213-216; April, pp. 277-280, 299.

ALDERSON, V. C. A directory of oil shale retorts, with bibliography. Colorado School of Mines Quarterly, vol. 15, Oct., 1920. Supplement. 21 pp. Data on the several retorts being built or operated experimentally or commercially in the U. S. and Canada. A supplementary bibliography of 4 pages is appended.

ALDERSON, V. C. Oil shale—a résumé for 1921. Colorado School of Mines, Circular of Information, 1922, 8 pp. A review of recent events in all producing countries. Concludes with a bibliography of recent articles.
See also Railroad Red Book, vol. 39, Jan., 1922, pp. 7-14.

ARON, A. L'industrie francaise des schistes bitumineux. Annales des Mines, 10th ser., vol. 9, 1906, pp. 47-75. Discusses the oil shale industry of France, giving statistics of production, 1893-1904. Also gives relation of French to Scotch retorting practice, as regards results obtained.

ASHLEY, G. H. Oil resources of black shales of the eastern United States. U. S. Geol. Survey Bull. 641, 1917, pp. 311-324. Published also as separate Bull. 641-L, 1917. Abstract in Jour. Wash. Acad. Sci., vol. 7, Nov. 4, 1917, pp. 564-565. Results of an investigation of shales in several eastern states to determine their content of oil and consequent commercial possibilities. Gives occurrence of the shale in these states, describes samples taken, and gives table of distillation tests of the samples.

BACON, R. F., and HAMOR, W. A. The American petroleum industry; with special chapters by F. G. Clapp, R. H. Johnson, J. P. Cappeau, and L. G. Huntley. New York, McGraw-Hill Book Co., 1916, 2 vols., 963 pp. Chap. 17 (pp. 807-814) on the shale-oil industry gives occurrence in the United States, history of the industry in Scotland, description of Scotch shales, mining methods, Scotch retorting and refining methods, products of Scotch shale oil and table showing yields of Scotch and other shales.

BACON, R. F., and HAMOR, W. A. Problems in the utilization of fuels. Jour. Soc. Chem. Ind., vol. 38, June 30, 1919, pp. 161T-168T. Discusses the importance of the conservation of fuels of all kinds, the need of further supplies of liquid fuel, the commercial status of the American oil-shale industry, and specific engineering problems in the utilization of fuels in various industries. See also Sci. Am. Suppl., vol. 88, Oct. 11, 1919, pp. 222-223. Extract in Railroad Red Book, vol. 37, Aug., 1920, pp. 723-725, 727.

BASKERVILLE, CHARLES. Value of American oil shales. Trans. Am. Inst. Min. and Met. Eng., vol. 65, 1921, pp. 229-232; discussion, pp. 233-236. Published first in Bull. Am. Inst. Min. and Met. Eng., June, 1919, pp. 957-960; discussion, December, 1919, pp. 3115-3118. Enumerates and discusses briefly the factors on which the economic success of the shale-oil industry depends; discusses the choice of suitable process of treatment and the urgent need of shale oil to meet the deficiency in fuel supply.

BEILBY, GEORGE. The production of ammonia from the nitrogen of minerals. Jour. Soc. Arts, vol. 33, 1885, pp. 313-325; discussion, pp. 326-327. Results of a study of Scotch shales.

BEILBY, GEORGE. Thirty years of progress in the shale oil industry. Jour. Soc. Chem. Ind., vol. 16, 1897, pp. 876-886. Discusses the evolution of the modern shale still and ammonia scrubber. Gives costs.

BOTKIN, C. W. A study of the saturated and unsaturated compounds of shale oils. Colorado School of Mines Quarterly, vol. 16, April, 1921, pp. 16-26. Railroad Red Book, vol. 38, May, 1921, pp. 603-609. Results of a study of shale-oil hydrocarbons to determine "the extent of their saturation, the cause of the unsaturates, the effect of cracking on saturation, and some of the properties of the unsaturated substances."

BOTKIN, C. W. A study of the saturated and unsaturated oils from shales. Chem. and Met. Eng., vol. 26, Mar. 1, 1922, pp. 398-401. Results of experimental work on the composition and properties of the lighter fractions and heavy undecomposed residues resulting from the pyrolytic treatment of crude shale oils.

BOWEN, C. F. Phosphatic oil shales near Dell and Dillon, Beaverhead County, Montana. U. S. Geol. Survey Bull. 661, 1918, pp. 315-320. Published also as separate Bull. 661-I, 1918. Gives stratigraphy and structure of the region and occurrence, properties, analyses and distribution of the shale.

CALDWELL, WILLIAM. The oil shales of the Lothians. Part II. Methods of working the oil shales. Memoir. Geol. Survey of Scotland. 1st ed., 1906, pp. 98-132; 2nd ed., 1912, pp. 95-135. Detailed discussion of methods of prospecting, mining, hauling and breaking shale.

CARNE, J. E. The kerosene shale deposits of New South Wales. Memoir. New South Wales Geol. Survey, Geol. No. 3, 1903. 333 pages. Maps, plates and sections. Appendix II. by W. R. Collis and R. G. Allman, "Mining titles for coal and kerosene shales." 2 pp. Describes development, geology, and method of treatment. Gives statistics of production, 1865-1901.

CARRUTHERS, R. G. The oil shales of the Lothians. Part I. The geology of the oil-shale fields. Memoir. Geol. Survey of Scotland, 2d ed., 1912, pp. 1-94. Based on work of H. M. Cadell and J. S. Grant-Wilson.

CHESNEAU, G. L'industrie des huiles de schiste en France et en Ecosse. Annales des mines, 9th ser., vol. 3, 1893, pp. 617-673. Outlines oil shale industry of France and compares distillation and refining practices of France with those of Scotland.

CONACHER, H. R. J. Oil shales and torbanites. Trans. Geol. Soc. Glasgow, vol. 16, part 2, pp. 164-192. Abstracts in Petroleum Rev., vol. 35, Dec. 23, 1916, p. 509. Geol. Mag., vol. 4, decade 6, February, 1917, pp. 93-94. Gives results of a microscopic study of the shales and torbanites of the Lothians to ascertain the substances in them that yield on distillation olefins, paraffins, and naphthenes in large proportions, whereas coal under similar treatment yields only tar containing small proportions of these compounds. Describes experiments and concludes that oil shales and torbanites are derived from the same substances as coal—vegetable matter—but that in the shales there has been a much more complete elimination of the woody matter, leaving the resins, cellulose and mud.

CONDIT, D. D. Oil shale in western Montana, southeastern Idaho, and adjacent parts of Wyoming and Utah. U. S. Geol. Survey Bull. 711, 1920, pp. 15-40. Published also as separate Bull. 711-B, 1919. Gives geologic sections of these districts, describes mode of occurrence of the shale and samples tested, and concludes with practical considerations on the technology of shale oil.

CRONSHAW, H. B. Oil shales, London, John Murray, Albermarle Street, 1921. 80 pp. Monograph prepared under the direction of the Mineral Resources Committee of the Imperial Institute. Describes oil shale and torbanite, the mining and distillation of oil shale, and the composition and properties of oil shale. Describes deposits of oil shale in various parts of the British Empire as well as those of other countries. Concludes with map showing the oil-shale deposits of the world and a bibliography.

CUNNINGHAM-CRAIG, E. H. The origin of oil shales. Proc. Roy. Soc. Edinburgh, vol. 36, 1916, pp. 44-86. Discusses relation of oil-rocks and oil-shales and the composition of kerogen and oil-shale. Concludes that oil-shale fields and oil fields are closely connected.

EGLOFF, GUSTAV, AND MORRELL, J. C. Supply of oil available from shales. Oil and Gas Jour., vol. 17, Aug. 9, 1918, pp. 46-48; Aug. 16, pp. 42, 44-46; Aug. 23, pp. 45-48. Discusses the relations of the various types of solids capable of giving up oil either by heat treatment or by the use of solvents; their possible mode of origin; their relation to other acid solid hydrocarbons and to petroleum; the economic distribution and treatment of oil shales; and the uses of their products. Gives numerous references to the literature on the subject. Also Railroad Red Book, vol. 35, Oct., 1918, pp. 13-19; Nov., pp. 13-20.

ELLS, R. W. Joint report on the bituminous or oil-shales of New Brunswick and Nova Scotia, also on the oil-shale industry of Scotland. Canada Department of Mines, Geol. Survey, 1909. 136 pages. Part I, Economics. Part II, Geology. Map No. 1181, issued separately in 1912, illustrates Part II. Discusses at length the oil-shale industry of Scotland. Describes geology and mining possibilities of oil shales of New Brunswick and Nova Scotia, giving results of distillation tests. French translation, 1914. 151 pp.

ELLS, S. C. Notes on the economic aspect of a Canadian oil shale industry. Canadian Chem. Jour., vol. 4, July, 1920, pp. 181-188. Statistics and suggestions relating to the development of an oil-shale industry in New Brunswick, with bibliography on oil shale.

FRANKS, A. J. A symposium of studies and researches in the oil-shale industry. Colorado School of Mines Quarterly, vol. 16, Oct., 1921. 76 pp. Reprints of papers, by A. J. Franks, as follows: Chemical engineering and economics in shale oil production, Chem. Age, vol. 29, Feb., 1921, pp. 67-69. Studies in Colorado shale oils, Chem. and Met. Eng., vol. 24, Mar. 30, 1921, pp. 561-564; vol. 25, July 13, 1921, pp. 49-53; Oct. 19, 1921, pp. 731-735; Oct. 26, 1921, pp. 778-783. Action of steam and gases on yields of ammonia from carbonization of shales and coal, Chem. and Met. Eng., vol. 23, Dec. 15, 1920, pp. 1149-1154. Introduction and bibliography by V. C. Alderson.

GAVIN, M. J. The necessity for research in the oil-shale industry. Chem. and Met. Eng., vol. 23, Sept. 8, 1920, pp. 489-495. Emphasizes the importance of continuous experimental work in connection with oil shales and of working out retorting and refining methods on a scientific basis in order to obtain maximum yields of products and the greatest possible profits. See also Science and Industry, vol. 2, Dec., 1920, pp. 746-760.

GAVIN, M. J. Possibilities of producing oil from oil shale. Bureau of Mines Reports of Investigations, October, 1920. 7 pp. Serial No. 2176. Abstract of paper before the Independent Oil Men's Association, September, 1920. Outlines briefly the methods practiced in Scotland and discusses the possibilities of American shales. See also Oil, Paint and Drug Rep., vol. 98, Oct. 18, 1920, Petroleum Sec., p. 12; Nat. Petroleum News, vol. 12, Oct. 6, 1920, p. 53. Petroleum Annual, 1921, Independent Oil Men's Association, Chicago, pp. 283-286, 288, 290.

GAVIN, M. J. The next mining problem: Proc. Am. Min. Cong., vol. 24, 1921, pp. 497-510. Discusses the problem of shale mining, describes Scotch methods, and emphasizes importance of attempts to reduce mining costs.

GAVIN, M. J. Past, present, and future of the oil-shale industry. California Oil World, vol. 13, May 26, 1921. Second section, pp. 84-85. Brief survey of the industry and its possibilities, including its growth and present status in France, Scotland, Australia and the United States; brief description of mining, retorting and refining methods (Scotch); and general discussion of the problems to be solved in the treatment of American shales for the maximum yield of valuable products.

GAVIN, M. J., AND AYDELOTTE, J. T. Solubility of oil shale in solvents for petroleum. Bureau of Mines Reports of Investigations, Jan., 1922. 4 pp. Mimeographed. Serial No. 2313. Gives method used and results obtained in experiments to determine the quantity dissolved from certain unheated shales by various solvents at or near the boiling points of these solvents. See also Petroleum Refiner, vol. 10, Jan. 19, 1922, p. 29. Oil Field Eng., vol. 24, Jan., 1922, pp. 20-21. Salt Lake Min. Rev., vol. 23, Feb. 28, 1922, pp. 17-18. Eng. and Min. Jour., vol. 113, Mar. 4, 1922, p. 369.

GAVIN, M. J., AND KARRICK, L. C. Nature of shale oil obtained from oil-shale assay retort used by the Bureau of Mines. Bureau of Mines Reports of Investigations, June, 1921, 11 pp. Mimeographed. Serial No. 2254. Describes method of distillation analyses used, and gives tables showing detailed results with shale oil from Scotland, shale oil from Scotch shale by the assay retort, shale oil from Soldier Summit, Utah, and Pennsylvania crude oil. See also Oil, Paint and Drug Rep., vol. 100, July 4, 1921, Petroleum Section, p. 8. Salt Lake Min. Rev., vol. 23, July 15, 1921, p. 17. Shale Rev., vol. 3, July, 1921, p. 5. Petroleum Times, vol. 6, July 23, 1921, p. 138.

GAVIN, M. J.; HILL, H. H., AND PERDEW, W. E. Notes on the oil-shale industry, with particular reference to the Rocky Mountain district. U. S. Bureau of Mines, Miscellaneous Publication, May 1, 1919. 51 pp. General treatise, with bibliography. Revised and issued June, 1921, as Bureau of Mines Reports of Investigations, Serial No. 2256, 36 pp. Mimeographed. General discussion of the subject, with short bibliography. Includes description of method and apparatus for assaying oil shale for oil yield, by L. C. Karrick; also methods for the laboratory distillation of oil shale for the determination of the available yield of crude oil and sulphate of ammonia obtainable therefrom, by E. M. Bailey.

GAVIN, M. J., AND SHARP, L. H. Some physical and chemical data on Colorado oil shale. Bureau of Mines Reports of Investigations, August, 1920. Serial No. 2152. 8 pp. Mimeographed. Gives data on the following physical and chemical properties of a representative Colorado shale. Weight per cubic foot of different sizes of shale; apparent specific gravity; specific heat of raw and spent shale; heat of combustion of raw and spent shale; thermal conductivity of raw shale; general analyses; oil yield; and heat of combustion of crude shale oil. Tables are also given comparing data on oil shale obtained in this investigation with similar data on other substances. See also Eng. and Min. Jour., vol. 110, Sept. 18, 1920, pp. 579-580. Oil and Gas Jour., vol. 19, Oct. 1, 1920, pp. 86-87. Gas Age, vol. 46, Sept. 25, 1920, pp. 219-220. Oil, Paint and Drug Rep., vol. 98, Sept. 13, 1920, Petroleum Sec., pp. 28-29. Oil News, vol. 8, Nov. 5, 1920, pp. 27-29.

GAVIN, M. J., AND SHARP, L. H. Short papers from the co-operative oil-shale laboratory. State of Colorado Co-operative Oil Shale Investigation Bull. No. 1, July, 1921. 68 pp. Papers are on the following subjects: (1) The fuel values of oil-shale and oil-shale products; (2) The nature and composition of shale gas; (3) Production tables and curves for shale oil as obtained from the horizontal rotary retort used in the Boulder laboratory; (4) The analytical distillation of shale oils; (5) Thermal calculations for the retorting of oil shales; (6) Tabulation of factors and formulae which have been found of value in oil-shale laboratories.

GARRETT, T. C., AND SMYTHE, J. A. The bases contained in Scottish shale oil. Part I, Jour. Chem. Soc., vol. 81, 1902, pp. 449-456; Part II, vol. 83, 1903, p. 763. A study of the nitrogen-containing constituents contained in shale oil, resulting in the recovery and identification of several homologues of pyridine from Scotch shale-oil.

GEORGE, R. D. Oil shales of Colorado. Colorado Geol. Survey Bull. 25, 1921. 78 pp. Gives geology of the Colorado oil shales and compares them as to occurrence, composition, oil-content, etc., with Scotch shales. Discusses retorting and refining problems, and describes the laboratory study of Colorado shales carried on by the Colorado Geological Survey.

GRAY, THOMAS. The phenols from shale oil. Jour. Soc. Chem. Ind., vol. 21, 1902, pp. 845-847. Experimental work on recovery of phenols from Scotch shale oils.

HAMOR, W. A., AND PADGET, F. W. The technical examination of crude petroleum, petroleum products and natural gas. New York, McGraw-Hill Book Co., Inc., 1920. 572 pp. A manual prepared for use of students of hydrocarbon chemistry and petroleum engineering, being arranged with a view to use as a laboratory companion to "The American Petroleum Industry", by Bacon and Hamor. A chapter (p. 183-198) on oil shales discusses the preliminary examination of shales, describes apparatus for field distillation, and describes method used at the Mellon Institute of Industrial Research for the assay of oil shales for yield of crude oil. Discusses also the examination of crude shale oil and suggests method of reporting results.

HENDERSON, N. M.; CRICHTON, A. H., AND BRYSON, JAMES. The history of shale retorts at Broxburn. Jour. Soc. Chem. Ind., vol. 16, 1897, pp. 984-992; discussion, pp. 992-993; vol. 18, 1899, pp. 246-248. Detailed descriptions and discussion of the Henderson, Philipstoun and Pumpherston retorts, including discussion of the effect of different retorts on the percentage and character of products obtained by the distillation of oil shales.

JONES, C. L. The problems of the American shale-oil industry. Chem. and Met. Eng., vol. 26, Mar. 22, 1922, pp. 546-553. "An outline intended to show briefly the present state of our knowledge in various divisions of the field of oil-shale technology and to suggest some channels which future investigational effort may take." Considers (1) problems for pure-science research, and (2) technologic problems.

JONES, J. R. The legal status of oil-shale deposits on the public domain. Eng. and Min. Jour., vol. III, Jan. 8, 1921, pp. 68-69. Gives provisions of the act of February 25, 1920, relating to oil-shale deposits and discusses requirements of the law.

KARRICK, L. C. A convenient and reliable retort for assaying oil shales for oil yield. Bureau of Mines Reports of Investigations, March, 1921. Serial No. 2229. Mimeographed. 7 pp. Revised April, 1922. Detailed description, with drawing.

KARRICK, L. C. Some factors affecting products from destructive distillation of oil shales. Bureau of Mines Reports of Investigations, Feb., 1922. 5 pp. Mimeographed. Serial No. 2324. Discusses influence of physical structure of shale on distillation products, effect of temperature lag, and effect of rate of heat supply.

LEWIS, J. O. Our future supplies of petroleum products. Bureau of Mines Reports of Investigations, October, 1920. 9 pp. Serial No. 2174. Address before the Independent Oil Men's Association, September, 1920. Discusses possibilities of increasing the supply of improved production methods, increased imports, use of vegetable and other substitutes, such as coal and oil shale, and by conservation in both production and use of crude oil and its products. See also Oil Trade Jour., vol. 11, Nov., 1920, pp. 96, 98, 126. Oil and Gas Jour., vol. 19, Oct. 15, 1920, pp. 74, 76. Oil, Paint and Drug Rep., vol. 98, Oct. 11, 1920, Petroleum Sec., p. 9. Min. Cong. Jour., vol. 6, Nov., 1920, pp. 582-586. Oildom, vol. 11, Nov., 1920, pp. 27-28. Petroleum Annual, 1921, Independent Oil Men's Association, Chicago, pp. 308, 310, 312, 314.

LUNT, H. F., and others. The oil shales of northwestern Colorado. Colorado State Bureau of Mines, Bull. No. 8, Aug. 1, 1919. 59 pp. A brief and non-technical summary of the most recent and authoritative information on oil shales, particularly the Colorado deposits, including description of the areas in Colorado in which shale occurs and general commercial considerations as to mining and refining of shale and marketing of its products. Discusses also method of acquiring oil shale lands, value of such lands, and mining regulations in the State. Concludes with bibliography. See also Nat. Petroleum News, vol. 11, Oct. 29, 1919, pp. 74-76, 78; Nov. 19, pp. 75-76, 78.

MCKEE, R. H., AND LYDER, E. E. The thermal decomposition of shales. I—Heat effects. Jour. Ind. and Eng. Chem., vol. 13, July, 1921, pp. 613-618. Gives origin and nature, and distribution of oil shales and discusses briefly their importance in the solution of the impending shortage of petroleum products. Describes experiments and investigations which have shown that the pyrobitumens do not decompose to form oils as a primary product of decomposition, but that the first substance produced is a heavy solid or semi-solid bitumen, and that the oils formed from the shale are the result of the decomposition or cracking of the heavy bitumen. Brief bibliography appended. II. Determination of the heat of reaction involved in their thermal decomposition. Jour. Ind. and Eng. Chem., vol. 13, Aug., 1921, pp. 678-684. Describes apparatus and method for determining this constant. Describes experimental procedure and results. Also published as Engineering and Scientific Paper No. 6 of Schools of Mines, Engineering and Chemistry, Columbia University, New York, August, 1921, 32 pp.

MILLS, E. J. Destructive distillation: A manualette of the paraffin, coal tar, resin oil, petroleum and kindred industries. 4th edition. London: Gurney & Jackson, 1892. 200 pp.

MORRELL, J. C., AND EGLOFF, GUSTAV. Destructive distillation of oil shales. Chem. and Met. Eng., vol. 19, July 15, 1918, pp. 90-96. Gives results of experimental distillation, describes products, and discusses the influence of sulphur, oxygen and nitrogen content, and the commercial possibilities of oil shale.

MORRELL, J. C., AND EGLOFF, GUSTAV. The economic position of oil shales. Met. and Chem. Eng., vol. 18, June 1, 1918, pp. 601-607. Economic distribution of oil shales, methods of treatment and comparative yields of products.

MURRAY, A. L. Sanitation in planning and developing oil-shale camps. Bureau of Mines Reports of Investigations, July, 1921. 7 pp. Mimeographed. Serial No. 2256. Prepared as part of paper "Use and supply of water for the oil-shale industry", by J. J. Jakowsky, to be published by the Department of Metallurgical Research, University of Utah, in co-operation with the U. S. Bureau of Mines.

RAILROAD RED BOOK. Progress in the American oil-shale industry. Vol. 38, Jan., 1921, pp. 11-21. Gives list of the more important processes for shale distillation and brief descriptions of fifteen experimental plants in the United States.

REDWOOD, BOVERTON. A treatise on petroleum. London, C. Griffith & Co., Ltd. 3rd ed., 1913. 3 vols. The shale-oil and allied industries, vol. II, Sec. VII, pp. 83-139. A general treatise on oil shale. Vol. III contains bibliography including references on oil shale. 4th edition revised; London, Charles Griffin & Co., 1921. 3 vols., 1350 pp.

REDWOOD, I. I. A practical treatise on mineral oils and their by-products. New York, Spon and Chamberlain, 1914. 336 pp. Includes a short history of the Scotch shale-oil industry, the geologic and geographic occurrence of Scotch shales, methods of treating the shales and refining the products.

ROBINSON, G. C., AND GOODWIN, W. L. New bases of the leucoline series, Part I, Trans. Roy. Soc. Edinburgh, vol. 28, 1879, p. 561; Part II, vol. 29, 1880, p. 265; Part III, vol. 29, 1880, p. 273. A study of the basic nitrogen compounds in Scotch shale oil, and identification of up to then unknown members of the leucoline (isoquinoline) series.

ROBERTSON, J. B. A chemical examination of organic matter in oil shales. Proc. Roy. Soc. Edinburgh, vol. 34, 1913-1914, pp. 190-201. Chem. Abs., vol. 9, 1915, p. 904. Describes experimental work and gives results.

ROWAN, F. J. On the physical conditions existing in shale-distilling retorts. Jour. Soc. Chem. Ind., vol. 10, 1891, pp. 436-443. Discussion of temperatures obtained in different shale retorts and their effect on the products.

SCHEITHAUER, W. Shale oils and tars and their products. Translated from the German by Charles Salter. London: Scott, Greenwood & Son, 1913. 183 pp. Gives geographic occurrence of the shale, its origin, properties and composition, and short history of the industry. Describes methods of treating, testing and refining, the products obtained, and their utilization.

SCHULTZ, A. R. Oil possibilities in and around Baxter Basin in the Rock Springs uplift, Sweetwater County, Wyoming. U. S. Geol. Survey Bull. 702, 1920. 107 pp. Gives stratigraphy and structure of the region and describes the oil-bearing rocks in detail, including oil-shale beds which are exposed.

SHARP, L. H., AND STRUNK, A. T. Some items of investment, expense and profit in commercial shale-oil production. Bureau of Mines Reports of Investigations, February, 1921. 3 pp. Mimeographed. Serial No. 2214. Outlines main items for large-scale operations under the following heads: Sources of income, capital investment, land, buildings, machinery and equipment, operating costs, selling and general expenses. See also Oildom, vol. 12, March, 1921, p. 47. Oil Trade Jour., vol. 12, March, 1921, p. 62. Chem. Age, vol. 29, Feb., 1921, pp. 69-70. Oil, Paint and Drug Rep., vol. 99, Feb. 21, 1921, Petroleum Section, p. 8.

STEUART, D. R. The oil shales of the Lothians. Part III. The chemistry of the oil shales. Memoir. Geol. Survey of Scotland, 1st ed., 1906, pp. 133-188; 2nd ed., 1912, pp. 136-194. Gives history of the oil shale industry, statistics of production in Scotland, and detailed discussion and data on the properties of various shales and their yield of products. Describes retorting and refining methods, the products of manufacture and their uses.

STRAHAN, A. Special reports on the mineral resources of Great Britain. Vol. VII. Lignite, jets, Kimmeridge oil-shale, mineral oil, cannel coals, natural gas. Part I. England and Wales. Mem. Geol. Survey, 1918. 66 pp., 2nd ed., 1920. Chapter II, on Kimmeridge oil-shales (pp. 24-40), gives their general distribution, uses and values, with analyses of several samples, and descriptions of occurrences in various localities.

THIESSEN, REINHARDT. Origin and composition of certain oil shales. Econ. Geol., vol. 16, June, July, August (Nos. 4 and 5), pp. 289-300. Part of a preliminary report on the microscopical study of oil shales undertaken in co-operation with the oil shale retorting experiments of the Bureau of Mines at Salt Lake City, Utah, and with the State Geological Survey of Kentucky. This is a detailed study and comparison of Devonian shales of Illinois, Indiana, Ohio, and Kentucky, with brief comparisons also with the shales of other geological formations and from other states and countries.

U. S. CONGRESS. House of Representatives. Committee on Public Lands. Mining of coal, phosphate, oil, etc. Report to accompany S. 2775. 66th Congress, 1st sess., 1919. House Report No. 398. 21 pp.

U. S. CONGRESS. Senate. An Act to promote the mining of coal, phosphate, oil, oil shale, gas, and sodium on the public domain. S. 2775, 66th Congress, Public, No. 146. Approved Feb. 25, 1920.

U. S. BUREAU OF MINES. Operating regulations to govern the methods of mining oil-shale, phosphate, sodium and potash, under the Acts approved February 25, 1920 (41 Stat. 437), and October 2, 1917 (40 Stat. 297), and the safety and welfare of the employees in connection therewith.

U. S. GENERAL LAND OFFICE. Regulations concerning oil shale leases, authorized by Act of February 25, 1920. Circular No. 671, 1920, 15 pp. Approved March 11, 1920. Explains qualifications of applicants, lands and deposits to which applicable, form and contents of application for lease, disposition of application, action on application, form of lease, and preferred right to a lease. Includes also text of the leasing Bill, S. 2775. See also Oil, Paint and Drug Rep., vol. 97, Mar. 22, 1920, Petroleum Section, pp. 11, 30. Shale Rev., vol. 2, April, 1920, pp. 5-6.

WILLIAMS, C. G. On the presence of pyridine among the volatile bases in the naphtha from the bituminous shale of Dorsetshire. Philos. Mag., 4th ser., vol. 8, 1854, pp. 209-212. Describes chemical work on pyridine in naphtha of shale oil of England.

WILLIAMS, C. G. On the volatile bases produced by destructive distillation of the bituminous shale of Dorsetshire. Jour. Chem. Soc., vol. 7, 1855, pp. 97-108. Data on analyses of shale oil.

WILLIAMS, H. E. Oil shales and petroleum prospects in Brazil. Trans. Am. Inst. Min. and Met. Eng., vol. 65, 1921, pp. 69-76; discussion, pp. 76-77. Published first as separate pamphlet, 1920, 8 pp. Abstract in Mining and Metallurgy, Sept., 1920, pp. 22-23. On the petroleum and shale resources of Brazil. Reprinted in Oil News, vol. 9, Feb. 5, 1921, pp. 36, 38, 40.

WINCHESTER, D. E. Oil shale in northwestern Colorado and adjacent areas. U. S. Geol. Survey Bull. 641, 1917, pp. 139-198. Published also as separate Bull. 641F, 1916. Jour. Wash. Acad. Sci., vol. 7, May 4, 1917, p. 265 (Abstract). With a view to showing the quantity and quality of the oil that may be distilled from the richer shale beds of this region, a study was made of its geology, and tests were made of the shale found. Numerous stratigraphic sections are given to show the character of the rocks exposed. Detailed description is given of the tests made, and the apparatus used, and results tabulated to show the yields of the different products.

WINCHESTER, D. E. Oil shale of the Uinta Basin, northeastern Utah, and results of dry distillation of miscellaneous shale samples. U. S. Geol. Survey Bull. 691, 1919, pp. 27-55. Published also as Bull. 691-B, 1918. Gives geology of the region, character of the shale, with results of distillation tests, properties and value of the shale oil obtained, and its probable origin. Results of dry distillation of samples from various states are given.

WINCHESTER, D. E. Oil-shales. Jour. Franklin Inst., vol. 187, June, 1919, pp. 689-703. Description of the occurrence and properties of the oil shales of the western United States and discussion of the present status and possibilities of the industry in this country. Reprinted in Railroad Red Book, vol. 36, Sept., 1919, pp. 695-697, 699-701, 703-704; October, pp. 767-769, 771-773, 775-776.

WOODRUFF, E. G., AND DAY, D. T. Oil-shale of northwestern Colorado and northeastern Utah. U. S. Geol. Survey Bull. 581, 1915, pp. 1-21. Describes occurrence of oil shales of Colorado and Utah, discusses their geology and gives results of distillation and analyses.

INDEX.